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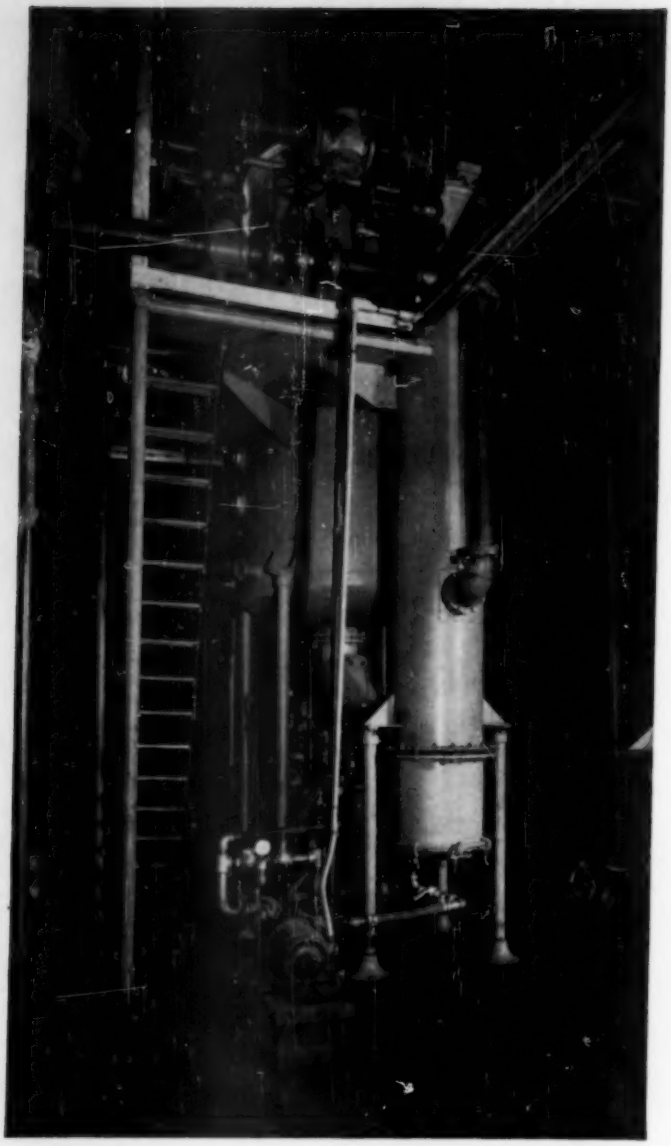

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Flavor Evaluations of Canned Fruits and Vegetables Treated With Newer Organic Insecticides[a,b]

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Several newer organic insecticides have been used in the entomology spray control program for four different vegetables (tomatoes, lima beans, carrots and potatoes) and two fruits (peaches and plums). These fruits and vegetables have been harvested at optimum stages of maturity, delivered immediately to the processing laboratory and canned according to accepted commercial practices. After a period of three months storage the samples have been evaluated for off-flavor by a panel of 12 previously trained tasters. The statistical analyses of the results show that the potato is the most susceptible to off-flavor imparted by the insecticides used in these experiments and that carrots and lima beans were the least susceptible. All of the benzene hexachloride formulations tested imparted an off-flavor to all of the fruits and vegetables except carrots. In addition, with some of the fruits and vegetables, chlordane, parathion, DDT and Toxaphene may impart an off-flavor to the edible part.

The use of the newer organic insecticides on fruit and vegetable crops *(4, 5, 6)* raises several important questions. Among the more important of these is the effect of the insecticides on the flavor imparted to the plant part which is to be marketed and consumed. Through cooperative efforts between the departments of Horticulture and Entomology it was possible to secure data on this question with a minimum of effort since experiments were already in progress to determine the effect of the various chemicals on the control of insects that attack fruit and vegetable crops. During the 1948 and 1949 season experimental plots provided sufficient material for processing and ultimate taste-panel testing of four different vegetables (tomatoes, lima beans, carrots and potatoes) and two fruits (peaches—Elberta and Welcome varieties—and plums).

A concept which has been fundamental to the program was that the use of an insecticidal material on a food crop which imparts an off-flavor to the edible part, regardless of how close the last application was made to the time of harvest, is of questionable value in the control program. Infestations of insects often develop on crops close to harvest time and the possibility that growers under such conditions might not follow the recommended procedures in applying insecticides, particularly with reference to the time of applications, is recognized.

SPRAYING AND HARVESTING PROCEDURES

Vegetables

The experiments were designed to provide a critical test of the effect of several insecticidal materials on the flavor of the edible plant parts. Consequently, the insecticides were applied at regular intervals throughout the growing season and relatively close to the date of harvest. Also, in certain instances materials were applied to crops contrary to recommended practices but this was part of the experimental plan concerned with the study of phytotoxicity and insect control.

The experimental vegetable plots were located at the Ohio Agricultural Experiment Station at Wooster in which nine insecticide formulations (parathion, tetraethyl pyrophosphate (TEPP), chlordane, benzene hexachloride (BHC), TDE (Rhothane), methoxychlor, DDT technical grade, DDT purified grade, and rotenone were compared in 1948 and thirteen insecticide formulations in 1949 (DDT technical grade, DDT purified grade, chlordane, TDE (Rhothane), chlorinated camphene (Toxaphene), lindane (99 percent gamma BHC), parathion, aldrin (Compound 118), methoxychlor, nitroparaffin propane base (CS 645A), nitroparaffin butane base (CS 674A), and rotenone). Each of the insecticides was applied to all vegetables in 4-fold randomized blocks. Zerlate (zinc-dimethyldithiocarbamate) was used in combination with all of the insecticides at the rate of 2 pounds in 100 gallons of spray mixture to provide protection against foliage diseases. The materials were applied at approximately weekly intervals with a Meyers Silver Prince wheel-barrow sprayer at the rate of 175 gallons per acre, except potatoes and lima beans which received the last application on August 10 and August 24, respectively. In the 1948 experiments, treatments 1 to 9 inclusive were applied on June 17 and 25, July 5, 13, 19 and 27, August 4, 9, 17, 24 and 31. Treatments 11 to 14 inclusive were applied to tomatoes growing in another series of plots on July 21 and 28, August 6, 12, 20 and 28, and September 4 and 10, except potatoes and lima beans which were given the final spray treatment on August 10. In the 1949 experiments the materials were applied to all crops included in the experiment on June 22, July 3, 11, 22, and August 1, 10 and 22.

The dates of harvest in 1948 were as follows: potatoes, October 12; carrots, October 25; Lima beans, August 31; tomatoes (treatments 1 to 10), September 1, and (treatments 11 to 14) on September 13. In 1949 the tomatoes were harvested on September 14; Lima beans on August 19; carrots on October 5, and potatoes on September 1.

At harvest time one-half bushel sample of tomatoes, lima beans, carrots and potatoes were taken from each of the four replicates for each treatment and these were composited into two samples by combining the sample harvested from two replicates of each treatment. Thus, two samples of vegetables harvested from plots treated with the various insecticides were available for processing. In harvesting the sample from each plot an attempt was made to pick vegetables of uniform quality (size, ripeness, etc.).

Fruits

The experimental plots of fruit were located at Wooster, Orrville, Berlin Heights and Port Clinton with eight insecticide formulations (benzene hexachloride, 10 percent low gamma (Lexone 10-G-W); benzene hexachloride, 12 percent; high gamma (Gam-Kil); benzene hexachloride, 25 percent; high gamma (Isotox); chlordane, 40 percent (Dowklor); DDT, 50 percent (Deenate 50-W); methoxychlor, 50 percent (Marlate 50); parathion, 25 percent (Thiophos 3422); compared in 1949. The 1948 data have been previously reported *(1)*. The treatments were applied to plots (four trees each) replicated four times. Applications were made with conventional high-pressure, hydraulic spraying equipment using a Bean single nozzle gun. Sprays were applied from the ground in order to

[a] Paper No. J23-50 Scientific Journal Series, Ohio Agricultural Experiment Station.

[b] Presented at the Tenth Annual Meeting of the I. F. T., Chicago, Illinois, May 24, 1950.

obtain adequate and thorough coverage of the fruit from all sides. The first application was made when jarring records indicated that hibernating adult curculios began to migrate into the experimental orchards and were continued through early June. Samples of the fruit were selected at harvest on September 2 on the basis of uniformity in maturity and freedom from insect injury. Although sampling was not strictly randomized, it was representative of different areas of the individual trees.

PROCESSING AND STORAGE

Immediately after harvest, the fruit and vegetable samples were delivered to the Horticultural Processing laboratory at Columbus where they were prepared for canning and subsequently processed according to acceptable commercial practices. In addition to the canning of all samples, one-half of each lot of lima beans were frozen, again according to acceptable commercial practices. After processing, the canned samples were stored at room temperature and the frozen samples at 0° F. (—18° C.) for 3 to 6 months prior to flavor evaluations.

FLAVOR EVALUATION METHODS

The taste panel used to evaluate these samples at the Ohio Agricultural Experiment Station (O.S.U.) was composed of teachers and graduate and undergraduate students of the Horticulture Products Division in the Department of Horticulture. Individuals were selected on the basis of (1) their threshold level and (2) their ability to repeat themselves on the four primary tastes, that is, sweet, sour, salty, and bitter. These values are graphically indicated in Figures 1 through 4. Each member selected (maximum of 14) was further trained by using known concentrations and dilutions of each insecticide to (1) familiarize the panel member with the specific insecticide and (2) to determine the percentage of the panel members able to detect the different concentrations of each insecticide. Table 1 summarizes some of the different insecticides evaluated with the percentage of the total tasters able to detect the specific insecticides.

After the storage period, two cans of each product were opened and prepared for the taste testing. The plums, peaches and tomatoes were served directly, that is, without heating. The carrots and lima beans were cooked in an open saucepan for 10 minutes, allowed to cool at room temperature and then served to the panel (10 members minimum at any one sitting). The panel members worked individually in booths and scored the samples from 0 to 10; 0 being unsatisfactory and 10 being perfect for flavor. The judging of each unknown sample was repeated at three different sittings by each panel member, thus making a minimum total of 30 ratings for any one sample.

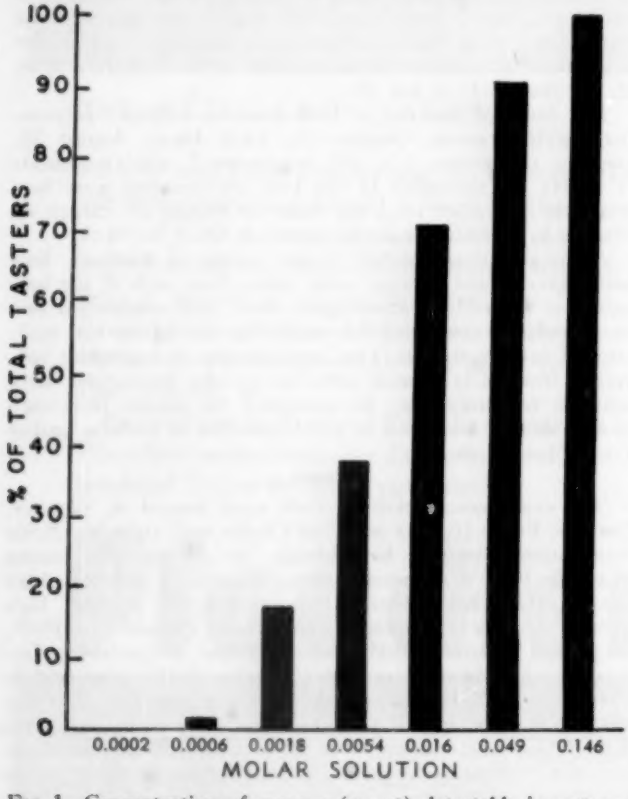

FIG. 1. Concentration of sucrose (sweet) detectable in water.

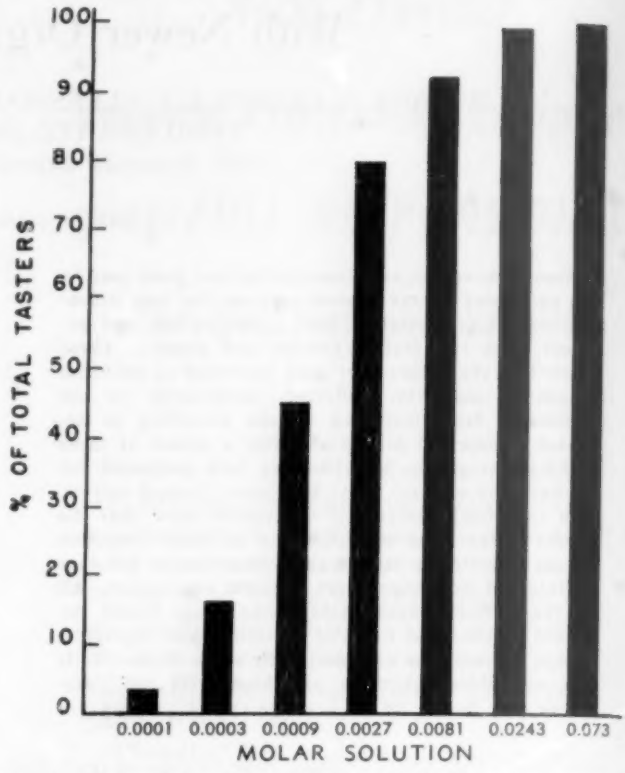

FIG. 2. Concentration of tartaric acid (sour) detectable in water.

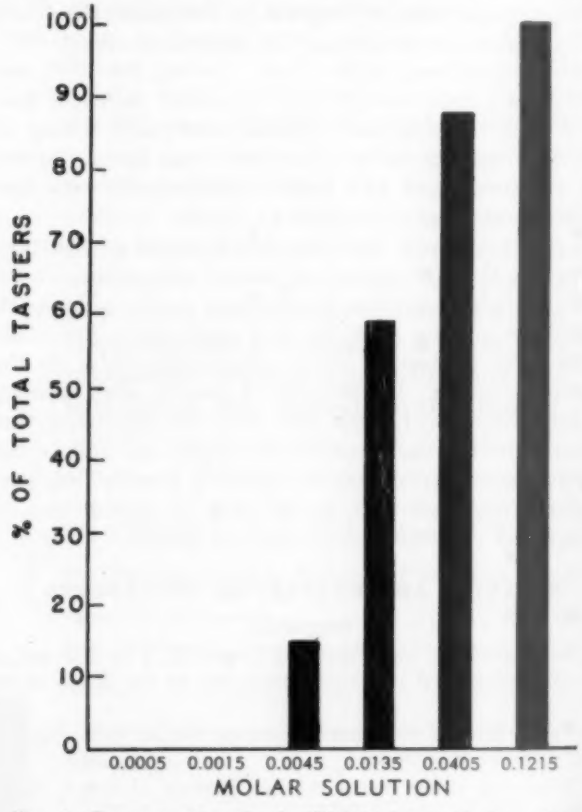

FIG. 3. Concentration of salt (NaCl) detectable in water.

TABLE 1

Concentration of Insecticide in Water Detectable to Taste Panel Members

	1 part in 100,000,000	1 part in 10,000,000	1 part in 1,000,000	1 part in 100,000	1 part in 10,000	1 part in 1,000	Not detectable [d]
	Percentage of tasters detecting off-flavor						
6 percent BHC (Lexone)	70	90	100	100	100	100	0
12 percent BHC (Penco W-12)	50	70	80	80	80	80	20
25 percent BHC (Isotox)	0	20	30	30	50	70	30
50 percent BHC (Dow C-814)	0	20	30	40	40	50	50
99 percent BHC (by recrystallization)	0	0	0	0	10	20	80
40 percent Chlordane (Dowklor)	0	20	30	30	40	60	40
25 percent Chlorinated camphene (Toxaphene)	0	10	20	30	50	50	50
25 percent Parathion (Thiophos)[c]	0	0	10	40	50	60	40

[c] Parathion tested by odor only. [d] Not detectable means the percentage of the total tastes unable to detect the insecticide in any of the concentrations used.

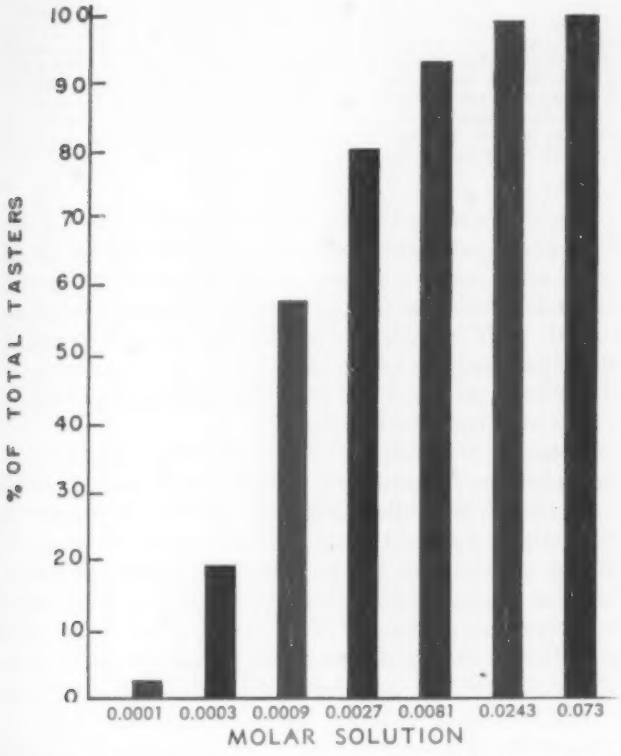

FIG. 4. Concentration of caffeine citrate (bitter) detectable in water.

The data were tabulated and then statistically analyzed using the analysis of variance method.

In addition to the 1948 and 1949 samples taste-tested at Ohio State University, samples of the 1948 processed vegetables were submitted to the National Canners Association (N.C.A.) and the Campbell Soup Company (C.S.C.) for their flavor-panel evaluations. The N.C.A. panels evaluated the samples "by comparing the treated samples against the untreated cooked sample of each lot. A score of 1 designated that a sample was normal, 2 designated a trace of off-flavor, and 3 a definite off-flavor" *(2)*. The C.S.C. panel evaluated the samples by scoring them on a scale from 0-100. They state that "in order to have some standard we have decided that the off-flavor is objectionable if over one-third of the panel noted it. Scores are then assigned on the basis of the percentage that fail to detect an off-flavor. If the score is below 66, we regard it objectionable" *(3)*.

The data obtained are given in Tables 2 to 8.

DISCUSSION

Table 1 show that the low gamma benzene hexachloride compounds imparted an off-flavor in concentrations as low as 1 part in 100,000,000; whereas high gamma benzene hexachloride compounds were not detectable to as many as 80 percent of the O.S.U. panel members in 1 part per 1,000. Chlordane, chlorinated camphene and parathion imparted specific tastes as evaluated by some panel members. Most of the panel members agreed that benzene hexachloride compounds imparted a musty flavor to the samples, but there was no agreement as to a good descriptive term for the other compounds.

TABLE 2

Effect of Insecticides on Flavor Contamination of Canned Potatoes. 1948 Samples.

Plot No.	Treatments	Formulae	Flavor Evaluations		
			C.S.C.	N.C.A.	O.S.U.
1	Parathion (25 percent) + Zerlate	2-2-100	33	1.7	9.00
2	TEPP (Nifos 40) + Zerlate	¼-2-100	100	1.5	9.11
3	Chlordane (Dowklor 50) + Zerlate	2-2-100	33	1.3	8.94
4	B H C (Dow C-814)[e] + Zerlate	1-2-100	0	2.4	4.78*
5	Methoxychlor (Marlate 50) + Zerlate	2-2-100	100	1.3	8.94
6	Rhothane 50 + Zerlate	2-2-100	17	1.4	8.61*
7	DDT (Deenate 50W) + Zerlate	1-2-100	67	1.2	9.11
8	DDT (Purified) + Zerlate	1-2-100	67	1.3	9.11
9	Rotenone 4 percent + Zerlate	5-2-100	67	1.5	8.83
10	Untreated check	.....	100	1.0	9.50
*Least significant difference at 0.01 level					0.67

[e] Formulated with BHC containing 99 percent of the gamma isomer.

TABLE 3

Effect of Insecticides on Flavor Contamination of Canned Carrots. 1948 Samples.

Plot No.	Treatments	Formulae	Flavor Evaluations		
			C.S.C.	N.C.A.	O.S.U.
1	Parathion (25 percent) + Zerlate	2-2-100	93	1.1	9.28
2	TEPP (Nifos 40) + Zerlate	¼-2-100	71	1.0	9.36
3	Chlordane (Dowklor 50) + Zerlate	2-2-100	93	1.1	9.21
4	B H C (Dow C-814)[f] + Zerlate	1-2-100	29	1.5	9.43
5	Methoxychlor (Marlate 50) + Zerlate	2-2-100	86	1.2	9.28
6	Rhothane 50 + Zerlate	2-2-100	93	1.1	9.57
7	DDT (Deenate 50W) + Zerlate	1-2-100	53	1.0	9.00*
8	DDT (Purified) + Zerlate	1-2-100	71	1.1	9.43
9	Rotenone 4 percent + Zerlate	5-2-100	39	1.8	9.57
10	Untreated check	.....	100	1.0	9.36
*Least significant difference at 0.01 level					0.33

[f] Formulated with BHC containing 99 percent of the gamma isomer.

TABLE 4

Effect of Insecticides on Flavor Contamination of Canned Tomatoes. 1948 Samples.

Plot No.	Treatments	Formulae	Flavor Evaluations		
			C.S.C.	N.C.A.	O.S.U.
1	Parathion (25 percent) + Zerlate	2-2-100	71	1.3	7.83
2	TEPP (Nifos 40) + Zerlate	¼-2-100	93	1.2	8.53
3	Chlordane (Dowklor 50) + Zerlate	2-2-100	57	1.8	8.57
4	B H C (Dow C-814)[a] + Zerlate	1-2-100	0	2.1	7.78*
5	Methoxychlor (Marlate 50) + Zerlate	2-2-100	82	1.4	8.19
6	Rhothane 50 + Zerlate	2-2-100	71	1.3	8.67
7	DDT (Deenate 50W) + Zerlate	1-2-100	36	1.5	8.78
8	DDT (Purified) + Zerlate	1-2-100	61	1.2	8.22
9	Rotenone 4 percent + Zerlate	5-2-100	100	1.5	8.83
10	Untreated check	.....	100	1.0	8.60
11	BHC (Lexone 50) 6 percent gamma	8-100	0	2.95	2.49*
12	BHC (Isotox) 25 percent gamma	2-100	0	2.5	5.55*
13	BHC (Dow C-814) 50 percent gamma	1-100	0	2.6	5.30*
14	Parathion	1 percent dust	14.2	2.4	6.89*
*Least significant difference at 0.01 level					0.80

[a] Formulated with BHC containing 99 percent of the gamma isomer.

TABLE 6

Effect of Insecticides on Flavor Contamination of Canned Vegetables. 1949 Samples.

Plot No.	Treatment	Formulae	Mean Flavor Evaluations		
			Potatoes	Tomatoes	Lima Beans
1	DDT technical 50 percent	1-100	7.71	8.43*	8.00
2	DDT purified 50 percent	1-100	7.64	8.46	7.65
3	Rhothane 50 percent	2-100	7.36	8.42*	7.80
4	Chlordane 40 percent	2-100	6.64	8.28	8.15
5	Toxaphene 25 percent	2-100	5.80*	8.09	7.85
6	Lindane 25 percent	1-100	4.28*	5.80*	4.50*
7	Parathion 25 percent	1-100	7.21	7.11*	8.05
8	Aldrin 25 percent	1-100	7.78	7.84	7.95
9	Methoxychlor 50 percent	2-100	7.36	8.07	7.45
10	Methoxychlor 50 percent	1-100	7.36	8.30	7.70
11	CS 645A 25 percent	1-100	7.36	8.45	7.80
12	CS 645A-CS 674A 25 percent	1/3-2/3-100	5.86*	8.20	7.80
13	Rotenone 4 percent	5-100	6.38	8.50	7.65
14	Untreated check	.....	7.14	8.15	7.85
*Least significant difference at 0.01 level			0.88	0.49	0.54

TABLE 7

Summary of Sprays, Schedules and Mean Flavor Scores on Stanley Prune-Type Plums. 1949 Samples.

Treatment [k]	Application [l] dates	Mean Flavor Evaluation Scores
1. BHC, 10 percent, low gamma	5-9, 5-17, 5-27, 6-8, 6-20	6.65*
2. BHC, 12 percent, high gamma	5-9, 5-17, 5-27, 6-8, 6-20	8.32
3. Chlordane, 40 percent [m]	5-9, 5-17, 5-27, 6-8, 6-20	8.72
4. Chlordane, 40 percent	5-9, 5-17, 5-27, 6-8, 6-20	7.78*
5. Parathion, 25 percent (1 lb.)	5-9, 5-17, 5-27, 6-8, 6-20	8.52
6. Parathion, 25 percent (2 lb.)[n]	5-9, 5-17, 5-27, 6-8, 6-20	8.35
7. Parathion, 25 percent (2 lb.)[n]	5-17, 5-27, 6-8, 6-20	8.55
8. Lead arsenate (used as check)	5-9, 5-17, 5-27, 6-8, 6-20	8.34
*Least significant difference at 0.01 level		0.55

[k] BHC, 10 percent low gamma used at the rate of 2½ pounds per 100 gallons; BHC, 12 percent at 3 pounds; Chlordane at 2½ pounds; Parathion at 1 and 2 pounds as noted above; Lead arsenate at 2½ pounds. Fermate added to each tank of spray at the rate of 1½ pounds per 100 for control of leaf spot. [l] May 9, petal-fall; May 17, shuck-fall; May 27, first cover; June 8, second cover; June 20, third cover; harvest date, August 24. [m] Armour's sticker was included in this schedule at 1 pint per 100 gallons. [n] Analysis of parathion residue on fruit from Treatment 5 showed 0.03 p.p.m. at harvest. Analysis of parathion residue on fruit from Treatment 6 showed 0.05 p.p.m. at harvest.

The data obtained by the three different panels on the 1948 processed vegetables could not be assembled on the same numerical scale because of variation in scoring methods. Both the C.S.C. and the N.C.A. panels presented their results in insufficient detail to permit statistical analyses of the data. The O.S.U. data were statistically analyzed and the least significant difference values are given in the tables.

Potatoes. In both the 1948 and 1949 samples BHC imparted an off-flavor to potatoes. This also was found to be true by both the C.S.C. and the N.C.A. panels. In 1948, the C.S.C. and O.S.U. panels found Rhothane to impart an off-flavor and in 1949 Toxaphene and a combination formulation of CS645A + CS674A were found to impart an off-flavor. The C.S.C. panel also found parathion and chlordane to give an off-flavor in 1948, although this was not detected by the N.C.A. or the O.S.U. panels in 1948 nor the O.S.U. panel in 1949.

Carrots. The carrot samples were the most difficult of all to judge to detect off-flavors. The N.C.A. panel found rotenone and BHC samples to give the poorest flavor. This was in agreement with the C.S.C. panel; however, the O.S.U. panel did not detect either of these samples as having off-flavor. The O.S.U. panel and the C.S.C. panel did score DDT (Deenate 50-W) as unsatisfactory for flavor on carrots. The 1949 data on carrots is not included in this report, but none of the

TABLE 5

Effect of Insecticides on Flavor Contamination of Canned and Frozen Lima Beans. 1948 Samples.

Plot No.	Treatment	Formulae	Flavor Evaluations			
			C.S.C.[i]	N.C.A.[i]	O.S.U.[i]	O.S.U.[j]
1	Parathion (25 percent) + Zerlate	2-2-100	100	1.5	8.4	9.3
2	TEPP (Nifos 40) + Zerlate	¼-2-100	100	1.4	9.0	9.6
3	Chlordane (Dowklor 50) + Zerlate	2-2-100	0	1.1	8.1	9.0
4	BHC (Dow C-814)[h] + Zerlate	1-2-100	0	3.0	3.9*	4.6*
5	Methoxychlor (Marlate 50) + Zerlate	2-2-100	100	1.1	9.1	9.2
6	Rhothane 50 + Zerlate	2-2-100	100	1.1	8.9	8.9
7	DDT (Deenate 50 W) + Zerlate	1-2-100	100	1.0	8.2	9.3
8	DDT (Purified) + Zerlate	1-2-100	100	1.0	9.1	9.3
9	Rotenone 4 percent + Zerlate	5-2-100	100	1.1	8.9	9.2
10	Untreated check	.....	100	1.0	7.8	9.5
*Least significant difference at 0.01 level					1.5	0.7

[h] Formulated with BHC containing 99 percent of the gamma isomer. [i] Canned. [j] Frozen.

TABLE 8

Summary of Sprays, Schedules and Mean Flavor Scores on Elberta and Welcome Peaches. 1949 Samples.

Figures refer to pounds of insecticide used in 100 gallons of water.

Treatment [a]	Petal Fall	Shuck Fall	First Cover	Second Cover	Third Cover	Fourth Cover	Fifth Cover	Mean Flavor Scores	
								Elberta	Welcome
Spray dates:	5/5	5/15	5/25	6/6	6/24	7/12	8/3		
1. Tech. BHC, 10 percent	2	2½	2½	2½	....	....	....	6.7*	7.4*
EPN, 35 percent	....	....	....	....	1½	1½	½		
2. Ref. BHC, 12 percent	1½	2	2	2	....	....	....	8.1	7.7
DDT, 50 percent	....	....	....	....	2	....	2		
3. Ref. BHC, 25 percent	0.8	1	1	1	....	....	....	8.4	8.5
DDT, 50 percent	....	....	....	....	1½	1½	1½		
4. Chlordane, 40 percent	2½	2½	2½	2½	....	....	....	8.7*	8.1
DDT, 50 percent	....	....	....	....	3	2	1		
5. Parathion, 25 percent	1	1	1	1	1	1	1	8.2	.....
6. Parathion, 25 percent	2	2	2	2	2	2	2	8.2	8.0
7. Methoxychlor, 50 percent	2	2	2	2	2	2	2	8.0	8.0
8. Lead arsenate	2	2	2	2	....	....	....	8.2	8.3
Zinc sulfate	4	4	4	4	....	....	....		
Hydrated lime	6	6	6	6	....	....	....		
DDT, 50 percent	2	2	2	2	2	2	2		..... [b]
9. Check	....	....	....	....	....	....	....	7.7	
*Least significant difference at 0.01 level								0.81	0.79

[a] Peach development: Full bloom, April 27; Half petal-fall, April 28; Two-thirds petal fall, April 29; Shuck-split, May 9; Shuck-fall, May 19; and Harvest date, September 2. [b] Treatment No. 8 used as check.

samples was found to have an off-flavor by the O.S.U. panel.

Tomatoes. All three panels agreed that the several BHC formulations used in the 1948 experiments imparted an off-flavor to tomatoes. In 1949 the O.S.U. panel again scored the BHC-treated samples as having an unsatisfactory flavor. The C.S.C. and N.C.A. panels found that the 1948 parathion dust-treated samples were off-flavor but the O.S.U. panel failed to detect taste contamination in these materials. The 1949 parathion-treated samples were rated as off-flavored by the O.S.U. panel. The C.S.C. panel also found that chlordane, DDT (technical), and DDT (purified) imparted an unsatisfactory flavor to the 1948 tomato samples.

Lima Beans. The 1948 samples of lima beans treated with BHC were found to be off-flavor by all three panels. In 1949 the O.S.U. panel again found that BHC imparted an off-flavor to this crop. Furthermore, the O.S.U. panel detected an off-flavor to the frozen product in the 1948 samples. In addition to the off-flavor found with BHC compounds the C.S.C. panel detected an off-flavor with chlordane-treated samples.

Fruits. The O.S.U. panel was the only panel used on the fruits. With plums both the low gamma BHC (10 percent) and chlordane (without the sticker) imparted an off-flavor to the fruit. Only the low gamma BHC (10 percent) imparted an off-flavor to the canned peach samples when using lead arsenate as a check; however if the actual check sample is used on the variety Elberta the chlordane treated sample gave a better flavor than the untreated check.

SUMMARY

In the experiments here reported the fact that serious insect infestations may develop on crops close to the harvest date is recognized. The insecticides were applied to vegetable crops at regular intervals throughout the growing season. Consequently, the last application to some crops was made relatively close to the time of harvest, later than it is common commercial practice to apply such insecticides.

Taste tests on the canned and frozen crops showed that the potato was most susceptible to off-flavors imparted by the insecticides used in these experiments while carrots and lima beans were the least susceptible. All of the low gamma or technical benzene hexachloride formulations tested imparted an off-flavor to all of the fruits and all of the benzene hexachloride formulations tested imparted an off-flavor to the vegetables except carrots. However, in 1948 the Campbell Soup Company and N.C.A. panels found that this latter compound did impart an off-flavor to even carrots.

Chlordane, parathion, DDT, Rhothane, and toxaphene also imparted an off-flavor to the edible plant part in some fruits and vegetables when used under the conditions of these experiments.

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On the Evaluation of Canning Processes

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A new definition of an "adequate process" is proposed and shown to lead to estimates of process times required which are greater than those deduced by the usual methods in common use at present. The difference is greater for products heating by convection than for those heating by conduction. The contribution of the cooling phase to the total lethal value of a process is calculated exactly for some particular cases and its importance is discussed. The principles on which a logical determination of safety factors might be based are discussed.

The heat treatment of canned foods must be sufficient to destroy organisms which could grow in the can and produce toxins dangerous to consumers or cause spoilage of the contents. The amount of heating required depends, therefore, on the resistance to heat of the bacterial spores which may be present in the pack, and on the rate of heat penetration into the contents of the can.

RESISTANCE TO HEAT OF BACTERIAL SPORES

The resistance to heat of bacterial spores is commonly specified by the thermal death time. This has been defined as the time required at a given temperature to destroy all the spores in a suspension of a standard number of spores in a standard medium.

Studies on the kinetics of spore destruction have shown that this simple definition of thermal death time is not strictly adequate. Experimental evidence indicates that the course of the destruction of spores at a constant temperature may be represented by the equation

$$c = c_0 e^{-kt} \qquad (1)$$

where c is the concentration of spores surviving at time t; c_0 is the concentration of spores at time $t = 0$; and k is a constant for a particular organism, medium and temperature.

This equation is supported by a great deal of experimental evidence but it is not yet certain that it is an adequate approximation under all conditions which might be encountered in canning practice.

In considering the sterilization of cans of food it is necessary to consider values of c less than 1.0. These are interpreted as probabilities of one spore surviving or the proportions of large numbers of similar samples identically treated which will contain survivors. Gillespy (*6*) has produced evidence of the validity of equation (*1*) down to values of c below 0.1, but it would be hardly practicable to check its validity experimentally for very much lower values of c.

It has been usual in the past to specify the resistance of spores by experimental values of the thermal death time determined directly. These are the times required to reduce a standard population to zero in a few samples of material, i.e. the final values of c are less than 1.0 but are not accurately determined. Alternatively the rate of decrease in spore numbers may be measured, and equation (*1*) used to deduce a thermal death time defined in terms of specified initial and final spore concentrations. Such a definition is necessary to give the term a precise meaning. It is, however, becoming common practice to specify the resistance of spores more directly in terms of equation (*1*) e.g. by the decimal reduction time, which is the time required to produce a tenfold reduction in population.

The rate of destruction of spores is a function of temperature and there is much experimental evidence to support the generally adopted assumption that the rate increases exponentially with temperature.

Equation (*1*) may be written

$$\frac{dc}{dt} = -kc \qquad (2)$$

Introducing the assumption that $\frac{dc}{dt}$ is an exponential function of temperature into equation (*2*) we have

$$\frac{dc}{dt} = -k_0 c e^{b(T-T_0)} \qquad (3)$$

where k_0 is a constant, being the value of the constant in equation (*2*) at the reference temperature T_0, T is the actual temperature, in general a function of position in the sample and time, and b is a constant for a particular organism and medium. The solution of equation (*3*) with $c = c_0$ when $t = 0$ is

$$c = c_0 \exp\left\{-k_0 \int_0^t e^{b(T-T_0)} dt\right\} \qquad (4)$$

or

$$\ln \frac{c}{c_0} = -k_0 \int_0^t e^{b(T-T_0)} dt. \qquad (5)$$

EVALUATION OF CANNING PROCESSES

Equation (*4*) may be written in the form

$$c_f = c_0 \exp\left\{-k_0 \int_0^{t_P} e^{b(T-T_0)}\, dt\right\}, \qquad (6)$$

where c_f is the concentration of spores at the end of the process, and t_P is the total process time. In general, c_f is a function of the position in the can, and the quantity $c_f dV$ may be termed the number of survivors in an element of volume dV. In this sense, the number of survivors in a can (n_f) is given by

$$n_f = \int c_f dV, \qquad (7)$$

the integral being taken over the whole volume of the can.

The quantity n_f is not, in general, a whole number and is usually less than 1. It must be interpreted in the same way as fractional concentrations i.e. as the average number of survivors in a large number of

identically treated cans or, if it is less than 1.0, it might be expressed as the probability of one spore surviving in a can. To avoid ambiguity it will be referred to as the *survival number for the can*. The ratio $\frac{n_f}{c_0}$ may be termed the *survival ratio for the can* and $\frac{c_f}{c_0}$ the *survival ratio for a point in the can.*

It is suggested that it is most logical to define an adequate process as one which will reduce the survival number for the can to some specified value.

The methods in general use at present consist essentially in evaluating the integral in equation (*6*) using temperature history data for the slowest heating point in the can. In the direct method of Bigelow et al. (*2*), the integral is evaluated graphically. Ball (*1a, 1b*) fitted equations to the relevant parts of the heating and cooling curves and derived formulae which he used to prepare tables and charts which permit rapid and easy calculation of solutions for particular cases.

These methods imply a definition of an adequate process, in the terminology suggested above, as one which will reduce the survival ratio at the slowest heating point of the can to some prescribed value. The difference in the consequences of this definition and the new one proposed above, is examined in later sections of this paper.

The new definition is expressed in terms of a survival number whereas that implicit in the usual methods is formulated in terms of a survival ratio. This change from a ratio to a number is convenient but not essential. Both definitions could be expressed in terms of either a survival number or a survival ratio. Whichever method of expression is used, the value assumed for c_0 must be specified in a complete statement of a problem. The essential distinction between the two definitions is that the new one is in terms of a survival number (or ratio) *for the whole can* whereas the old one is in terms of the survival ratio *at the slowest heating point.*

Stumbo (*9*) has pointed out that with a uniform spore distribution in the can, the number of spores actually subjected to the conditions at the slowest heating point will be very small. Some regions in the can receiving slightly more severe heat treatment will be relatively large in volume and so contain more spores. The chance of survival of a spore in such a region may therefore be greater than at the slowest heating point. He proposes to base estimates of the sterilizing value of processes on the chance of survival of a spore in the region of the can for which this is greatest. This is equivalent to defining an adequate process in terms of survival numbers in regions of the can whose volumes vary with distance from the center. In a later paper (*10*) he has implicitly suggested a definition similar to the new one proposed above.

APPLICATION TO PRODUCTS HEATING BY CONVECTION

To explore the practical implications of equation (*7*), consider first the ideal case of a can heated uniformly and instantaneously to retort temperature and cooled instantaneously after a heating period t. For this case equation (*7*) reduces to

$$n_f = c_0 V \exp.\{-k_0 t\, e^{b(T-T_\theta)}\}, \qquad (9)$$

where V is the volume of the can. If $T = T_\theta$, i.e. retort temperature is selected as the reference temperature T_θ, this becomes

$$n_f = c_0 V e^{-k_0 t}. \qquad (10)$$

This ideal case is of interest as a crude approximation to the actual temperature history in a product heated by convection. Equation (*10*) shows that the survival number for the can is proportional to the volume of the can. Of more direct interest is the time of heating necessary to reduce n_f to some specified value. This is given by

$$t_1 = \frac{1}{k_0} \ln \frac{c_0 V}{n_f} \qquad (11)$$

$$= \frac{2.3026}{k_0} \log_{10} \frac{c_0 V}{n_f}. \qquad (12)$$

Take 250°F. (121.1°C) as the reference temperature T_0 and assume we have to deal with an organism such that an initial population of 10^{10} spores/ml. is reduced to 1 spore/ml. in 2.78 minutes at 250°F. (These figures may be regarded as applying to a hypothetical organism, similar in resistance to the strains of *Cl.botulinum* studied by Esty and Meyer (*4*)). For this case

$$k_0 = 8.28 \text{ (min.)}^{-1}$$

and from (*12*)

$$t_1 = 0.278 \log_{10} \frac{c_0 V}{n_f} \text{ minutes.}$$

Using this equation we deduce the figures shown in Table 1.

As already indicated, the figures in this table are for an ideal case and do not apply exactly to any actual canning process. It is evident that under the conditions assumed, a slightly greater process would be required for No. 10 cans than for No. 2 but the difference is small. The time of heating required varies more with the level of initial contamination assumed, but the levels are widely different (successive ratios of 1000 to 1).

In an actual canning process with convection heating material, an appreciable time is required to heat the can contents to retort temperature, this time varying with the can size as shown by Schultz and Olson (*8*). There is, too, an appreciable time lag in cooling the contents of the can at the end of the heating process.

Another factor to be considered is the movement of spores from place to place in the can during processing. Each spore is subjected to some sort of average of the temperature history of different points in the can. In a purely liquid pack a good approximation to the actual chances of survival of a spore would probably be given by substituting $\overline{T}$, the average temperature in the can at a particular time, for T, the temperature at a particular point in the can, in equation (*8*) giving

$$n = c_0 V \exp\left\{-k_0 \int e^{b(\overline{T}-T_0)}\, dt\right\}. \qquad (13)$$

The figures in Table 1 will apply to this case if the times at 250°F. are interpreted as equivalent times at 250°F. calculated in the ordinary way for the *average* temperature history of points in the can.

For products such as peas, asparagus, etc., where it is likely that spores may remain on the surfaces of pieces of the vegetable,

a better approximation would be given by the direct evaluation of the integral in equation (*8*), i.e. the survival ratios for particular positions should be calculated and then summed, instead of averaging the temperature histories.

There are not enough detailed experimental data available to investigate the numerical differences which would result from these two methods of averaging conditions, but from consideration of the equations, and the data of Jackson and Olson (*7*), it is fairly evident that for the case of spores in fixed positions, the survival number for the can will be only slightly different from the case of perfect mixing of the spores. Table 1 should, therefore, provide reasonably accurate estimates for this case when the times are interpreted as equivalent times at 250°F. for the average temperature history in the can. If, as is usual, the temperature at the slowest heating point of the can is the only one measured, this could be used but the error involved in using Table 1 would be somewhat greater.

An important practical implication of these considerations is shown by comparing the figures in the last line of Table 1 with the others. The figures in the last line are approximately those which would be deduced by the older methods of process evaluation. The method suggested here leads to the conclusion that for a No. 2 can the equivalent time at 250°F. required for a specified degree of safety is approximately 0.8 minutes longer than that indicated by the older methods. For a No. 10 can the difference is approximately 1.0 minutes. These calculations are based on a hypothetical strain of *Cl. botulinum*. Calculations based on other organisms would give different results, but the effect of can size on the process required and the difference in the consequences of the old and new definitions would be similar.

PRODUCTS HEATING PURELY BY CONDUCTION

A good deal of the destruction of spores near the center of a can of solid material occurs after cooling of the surface has begun; the maximum center temperature is reached a few minutes after the steam is cut off. Temperature histories during both the heating phase and the cooling phase are therefore required.

Consider a cylinder of diameter 2a and height 2L, composed of uniform material of thermal diffusivity α. Let the initial temperature be T_1 throughout and the surface held at T_2 from $t = 0$ to $t = t_1$, and T_3 thereafter. For $t < t_1$, i.e. during the heating phase, the temperatures in the can are given by (*3*)

$$T - T_1 = (T_2 - T_1)\,\phi\,(r, x, t), \qquad (14)$$

where

$$\phi\,(r, x, t) = 1 - \frac{8}{\pi}\sum_{n=1}^{\infty}\frac{J_0\left(\beta_n \frac{r}{a}\right)}{\beta_n J_1(\beta_n)} \cdot e^{-\beta_n^2 \pi^2 \alpha t/a^2} \cdot \sum_{m=0}^{\infty}\frac{(-1)^m}{2m+1}\cos\frac{(2m+1)\pi x}{2L} \cdot e^{-(2m+1)^2\pi^2\alpha t/4L^2} \qquad (15)$$

and β_n is the n^{th} positive root of $J_0(\beta) = 0$.

For $t > t_1$ it is shown in appendix 1 that

$$(T - T_1) = (T_2 - T_1)\,\phi\,(r, x, t) + (T_3 - T_2)\,\phi\,(r, x, t - t_1). \qquad (16)$$

In using these equations for canning calculations it is usually sufficient to retain only the first term of each of the series in $\phi\,(r, x, t)$ but a number of terms must be retained to obtain adequate accuracy in $\phi\,(r, x, t - t_1)$. For small values of $(t - t_1)$ it is almost essential for numerical work to replace the series by the corresponding expansions in terms of error functions (*3*), which are more rapidly convergent.

To evaluate a process it is necessary to substitute the temperature histories of equations (*14*) and (*16*) in equation (*6*) and evaluate the integral of equation (*7*). To calculate c_f at a particular point in the can either graphical or arithmetical methods may be used. In this work the well known graphical methods were used for checking the results but most reliance was placed on arithmetical methods.

If only one term is retained in each series in $\phi\,(r, x, t)$, equation (*14*) may be rewritten:

$$T = T_2 - A\,e^{-\gamma t}, \qquad (17)$$

where $A = \frac{8}{\pi} \cdot \frac{T_2 - T_1}{\beta_1 J_1(\beta_1)} \cdot J_0\left(\beta_1 \frac{r}{a}\right) \cdot \cos\frac{\pi x}{2L}$

and $\gamma = \alpha\left(\frac{\beta_1^2}{a^2} + \frac{\pi^2}{4L^2}\right)$.

Substituting in equation (*5*), the destruction of spores during the heating phase is given by

$$\ln\frac{c}{c_0} = -k_0\int_0^{t_1}\exp b(T_2 - T_0 - Ae^{-\gamma t})\,dt = \frac{k_0}{\gamma}e^{b(T_2 - T_0)}\left\{\mathrm{Ei}(-Ab\,e^{-\gamma t_1}) - \mathrm{Ei}(-Ab)\right\}. \qquad (18)$$

It is often convenient to express the results in terms of F, the equivalent time at 250°F. (121.1°C.), rather than $\ln\frac{c}{c_0}$. If T_0 is taken as 250°F.,

$$F = \frac{1}{\gamma}e^{b(T_2 - T_0)}\left\{\mathrm{Ei}(-Ab\,e^{-\gamma t_1}) - \mathrm{Ei}(-Ab)\right\}. \qquad (19)$$

Calculations have been made for a No. 2½ size can filled with a solid material of a thermal diffusivity 0.090 cm.²/min. assuming it is a cylinder 10.00 cm. in diameter and 10.16 cm. high. As before, b was taken as 0.1279(°F.)$^{-1}$ i.e. in the usual terminology of canning technology, $z = 18$°F. Equation (*19*), and tables of exponential integrals (*5*) were used for calculating F values for the heating phase at 49 positions in the can. For the cooling phase values of the integrand of equation (*6*) were calculated from equation (*16*) for times at intervals of one minute, and the integrals evaluated by Simpson's rule. For the smaller values of $(t - t_1)$, the series in the second term of equation (*16*) were replaced by the more rapidly convergent expansions in terms of error functions[b] (*3*). One calculated temperature history is shown in Fig. 1. An experimental curve

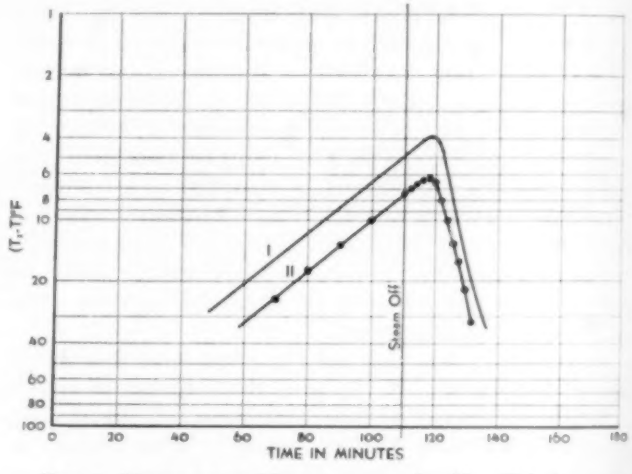

FIG. 1. Temperature history curves for No. 2½ size cans. Upper curve: Calculated center temperatures for case considered in the text. Lower curve: Experimental values for a can of pumpkin (initial temperature approximately 140°F. (60°C.).

[b] The error function expansion for the "cylinder term" is useful only when $(t - t_1)$ is small and $\frac{r}{a}$ is not small (*3*), but it is profitable to use it for the first 3 or 4 minutes of cooling when the value of $\phi\,(r, x, t - t_1)$ remains negligible for the smaller values of $\frac{r}{a}$. For larger values of $(t - t_1)$ the series for the cylinder term in equation (*15*) is not unduly cumbersome.

for pumpkin in a No. 2½ size can is plotted on the same diagram. This is representative of a large number of observations on solid packs in this laboratory and is clearly very similar in form to the calculated curve. The temperatures plotted in curve II were measured with a 30 swg enamelled copper-constantan thermocouple, with the reference junction in melting ice. A sensitive potentiometer permitting readings to within 0.1°F. was used. Frequent readings were taken in the early stages of cooling.

The results of one set of calculated F values are shown in Tables 2, 3 and 4.

To calculate the survival number for the can from the data in Tables 2, 3 and 4, we assume, as before, that we have to deal with organisms such that the population is reduced by a factor of 10^{10} by heating for 2.78 minutes at 250°F. (121.1°C.). We have $k_0 = 8.28$ (min.)$^{-1}$.

or
$$\frac{c}{c_0} = 10^{-10F/2.78}.$$

Using this last relationship, the figures in Table 5 were deduced from those in Tables 2, 3 and 4. These are, of course, based on the total F values (heating phase plus cooling phase). From this table it appears that there is, for each length of process, a roughly ellipsoidal region in the center of the can, in which the value of the process varies only slightly (e.g. by a factor of less than 10 in a quantity of the order of 10^{20}). Outside this region the survival ratio decreases sharply. This result is clearly due to compensating effects of lag in heating and lag in cooling in the central region.

For the can under consideration equation (7) may be written

$$\begin{aligned} n_f &= 4\pi \int_0^a \int_0^L r\, c_f\, dx.dr. \\ &= 4\pi a^2 L \int_0^1 \int_0^1 \frac{r}{a} c_f\, d\left(\frac{x}{L}\right) d\left(\frac{r}{a}\right) \\ &= 2V \int_0^1 \int_0^1 \frac{r}{a} c_f\, d\left(\frac{x}{L}\right) d\left(\frac{r}{a}\right). \end{aligned} \qquad (20)$$

The integrations of equation (*20*) were carried out graphically For the cook of 110 minutes it was found that

$$\frac{n_f}{c_{f_0}} = 0.0168V = 13 \text{ ml. (approx.)}$$

where c_{f_0} is the final concentration at the center.
Corresponding values for the cooks of 100 minutes and 90 minutes are 14 ml. and 15 ml. respectively.

CONCEPT OF EQUIVALENT VOLUME

The survival number for the can considered in the previous section, for a cook of 110 minutes, is numerically equal to 13 times the final concentration at the center, i.e. it is equal to the survival number for 13 ml. of material subjected to the conditions at the center. The can may be said, therefore, to have an equivalent volume (v) of 13 ml. The equivalent volume is then defined as the ratio of the survival number for the can to the final concentration at the center:

$$v = \frac{n_f}{c_{f_0}} \qquad (21)$$

The value of c_{f_0} can be determined from temperature measurements at one point, and if estimates of v are available, n_f or a suitable related quantity can be calculated by a simple and obvious modification of the older methods of process evaluation. For example, if F_e is the F value required to reduce the survival ratio at the center to some prescribed value, the F value at the center required to reduce the survival number for the can to the same prescribed value is $F_e + 0.278 \log_{10} v$, for the organism considered here.

VARIATION OF EQUIVALENT VOLUME WITH PROCESSING CONDITIONS

The concept of equivalent volume will be a useful one only if it is fairly easy to obtain estimates of its value of adequate accuracy for practical purposes. The equivalent volume must vary with processing conditions, but if this variation is small compared with other inevitable uncertainties in the data used in calculations (e.g. initial spore load), it will be possible to obtain estimates of v which are accurate enough for practical purposes from a relatively small number of detailed studies of particular packs.

Consequently some exploratory studies of the variation in v with some processing variables have been carried out. The results are summarized in Fig. 2.

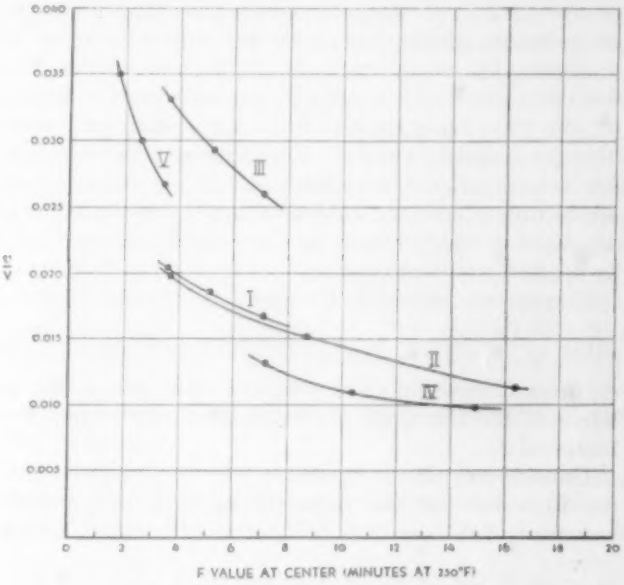

FIG. 2. Variation in equivalent volume with processing conditions. All curves are for cans of material of thermal diffusivity 0.090 cm²./min.; retort temperature 240°F. (115.6°C.); cooling water 70°F. (21.1°C.); $z = 18$°F. Curve I. No. 2½ size can; initial temperature 180°F.; 2.78 minutes at 250°F. (121.1°C.) required to reduce spore concentration by a factor of 10^{10}. Curve II. As for curve I except initial temperature 100°F. Curve III. As for curve I except 5 minutes at 250°F. required to reduce spore concentration by a factor of 10^{10}. Curve IV. As for curve I except can dimensions increased in ratio 2:1 (comparable with No. 10 size can). Curve V. As for curve I except can dimensions reduced in ratio 2:1 (comparable with No. 8Z size tall can).

In this diagram $\frac{v}{V}$ is plotted against F value at the center of the can using a z value of 18°F. Each curve connects points for processes differing only in length of cook.

It is clear from all the curves that v decreases with increasing length of cook. For F_e values at the center above about 3 minutes, the rate of change in v is quite small. For small values of F_e the value of $\frac{v}{V}$ rises rather steeply; on theoretical grounds it would be expected that the limiting value of $\frac{v}{V}$ as $F \to 0$ would be 1.0.

Comparing curves I and II it is seen that v varies with the initial temperature but the effect is small.

Comparing curves I and III it is clear that an increase in resistance to heat of the spores, increases the equivalent volume. Increasing the F value required to reduce the population by a factor of 10^{10} from 2.78 min. to 5 min. gives an increase in equivalent volume of roughly 50%.

To study the effect of varying the size of can but not the shape, we note that in equation (*16*) $(T - T_1)$ may be regarded as a function of $\frac{t}{a^2}$, since the ratio $\frac{a}{L}$ remains constant if the shape is unchanged. Consequently if T is the temperature at time t at a position defined by values of $\frac{r}{a}$ and $\frac{z}{L}$, in a can of diameter a, it will also be the temperature at the corresponding point in a can of diameter λa at time $\lambda^2 t$. Thus the temperature history curve for the center of a can of diameter λa for a cook of $110\lambda^2$ minutes can be derived from Fig. 1 simply by multiplying the figures on the time scale by λ^2. The same relations clearly hold for lethality curves. Consequently the F values for a cook of $\lambda^2 t_1$ minutes in a can of diameter λa are λ^2 times those for a cook of t_1 minutes in a can of diameter a. Differences in thermal diffusivity could be studied in a similar way.

Comparing curves I, IV and V it is seen that the ratio $\frac{v}{V}$ decreases with increasing can size. The difference between an 8 size tall can and a No. 10 size can for the same F_e value is of the order of a factor of 2.

Considering all the curves in Fig. 2, it appears that for F_e values of the order of magnitude generally recommended (say 3–6 min.) the extreme variation in $\frac{v}{V}$ is of the order of a factor of 2. The survival number for the can is proportional to $c_o v$, and as a rule, the uncertainty in c_o is unevitably much greater than a factor of 2 (perhaps a factor of 100 or 1000). It would be reasonable, therefore, to regard an estimate of $\frac{v}{V}$, which is trustworthy to within a factor of 2 as near enough for practical purposes, and a value of 0.02 or 0.025 might be adopted for solid packs in cans of roughly the same shape as a No. 2½. It should be emphasised that this might be misleading for cooks very much below the usual *Cl. botulinum* standards.

For the ideal case of heating by convection discussed above, the equivalent volume is V the total volume. For actual packs heating by convection the true equivalent volume is presumably less than V but of the same order of magnitude i.e. the difference between the consequences of the new definition of an adequate process proposed here and the older definition is much greater for products heating by convection than for those heating by conduction.

IMPORTANCE OF THE COOLING PHASE

Tables 2–4 show the relative importance of the heating and cooling phases in a product heating by conduction. As the length of process is increased the relative importance of the cooling phase decreases though its absolute value increases. It is well known that in products heating by convection, the amount of spore destruction during the cooling phase is very small (F value less than 5 percent of that for the heating phase).

It is fairly common practice to leave the cooling phase out of account in evaluating processes, regarding it as a useful safety factor. For a product heating by convection, this safety factor is less than 1.05 on the F scale or less than about 3 on the chance of survival scale. Near the center of a No. 2½ can heating by conduction the sterilizing value of the cooling phase is about 2 minutes on the F scale, or a reduction in the chance of survival of a spore of the order of 10^7 i.e. neglect of the cooling phase introduces a safety factor of less than 3 for a product heating by convection and one of the order of 10^7 for a product heating by conduction in a No. 2½ size can.

The sterilizing value of the cooling phase in a product heating by conduction varies substantially with the size of can.

POSITION OF MINIMUM F VALUE IN PRODUCTS HEATING BY CONDUCTION

In Tables 2, 3 and 4, the total F values show minima at positions slightly displaced from the center of the can. However the uncertainty of the calculated figures is of the order of 0.01 minutes so that it is not proved beyond doubt that the center is not really the point of minimum F value in this case. It would not, however, be surprising if the apparent displacement of the minimum is real; if the initial temperature were equal to retort temperature the minimum would occur where cooling is fastest i.e. on the surface of the pack. It seems, therefore, theoretically possible to have the minimum anywhere in the can by using high enough initial temperatures. It is also possible that the minimum would be substantially displaced with very short cooks.

The evidence available so far indicates that although the center of the can is not necessarily the point of minimum F value, the value at the center is very close to the minimum under normal processing conditions. If this conclusion is sound the exact location of the minimum in any particular case is only of academic interest.

COMPARISON WITH BALL'S MATHEMATICAL METHOD

In Ball's nomenclature the constants for the cases considered in Tables 2, 3 and 4 are f = 78.3; j = 2.04; z = 18°F.; RT = 240°F; IT = 180°F; m + g = 170°F.

The F values at the center for the heating phase, calculated by his methods (*1b*), agree exactly with those given in Tables 2, 3 and 4. The total F values are, however, widely different. For cooks of 110, 100 and 90 minutes the total F values given by Ball's mathematical method are 5.21, 3.63 and 2.36 respectively compared with 7.05, 5.24 and 3.71 obtained by the present methods. Ball has pointed out that the form of cooling curve assumed in his analysis is not always a good approximation to experimental curves and for such cases he recommends the use of the direct method or his own arithmetical modification of it (*1b*). It appears that all products heating by conduction should be included in this category. The relation between the true cooling curve and the form used by Ball is discussed in Appendix 2.

MAGNITUDE OF APPROPRIATE SAFETY FACTORS

The values adopted for the initial and final numbers of spores in the above discussion are all purely arbitrary. This does not affect the present argument because the difference between the estimates of the equivalent time at 250°F. (121.1°C.) required for an adequate process, obtained by the two methods, is almost independent of the actual limits.

For the calculation of the process time required for a particular cook, it is, of course, necessary to adopt appropriate limits. The usual methods of process evaluation based on Esty and Meyer's data for *Cl. botulinum* involve the adoption of a ratio $\frac{n_f}{c_0}$ of the order of 10^{-12}, though there are large variations in the actual value between recommended processes for different materials. No attempt will be made here to decide what are the most appropriate limits to adopt for any particular packs but some of the principles on which a decision might be based will be discussed briefly.

If n_f is considered as an independent quantity, its value should vary with the organisms under consideration. If the organisms likely to survive could cause spoilage but not endanger the health of consumers, e.g. flat sour organisms, a relatively large value of n_f would be appropriate, say 10^{-2} or 10^{-3}. If, however, there is risk of a highly toxic organism such as *Cl. botulinum* surviving, it would be necessary to adopt a very much lower value for n_f.

Estimates of c_0 can be obtained from bacteriological studies of material in canneries. However, it is extremely difficult to obtain a precise estimate of c_0 because of the great variation in the numbers and types of organism in different parts of one batch of raw material and between different batches.

There are strong arguments in favour of adopting for c_0 the maximum likely value. It is theoretically possible to deduce an estimate of any desired precision of an appropriate maximum value of c_0 by statistical analysis of the results of bacteriological study of an adequate number of properly selected samples of material. Such an estimate can only be properly specified by quoting both the value and the probability of its occurrence i.e. the actual value cannot be determined without adopting an arbitrary standard of safety.

In practice it is unlikely to be generally feasible to obtain the data necessary for a precise determination of the appropriate value of c_0 by statistical methods. It is then necessary to decide on a value using less definite evidence. In this case, too, some safety factor must be allowed for in the estimate of c_0.

In considering safety factors it is necessary to take into account likely variations in retort temperatures, timing errors, and other physical factors as well as purely bacteriological questions. It is likely to be profitable to express these in terms of their effect on the ratio $\frac{n_f}{c_0}$. Proper allowance can then be made for these physical variables by modifying the estimate of an appropriate ratio derived by considering n_f and c_0 from the bacteriological point of view.

The order of magnitude of the effects of some factors on estimates of the survival number for the can are given in Table 6. Some of the figures in this Table are very rough estimates and some apply to a set of conditions not completely specified, but they are sufficient to show the relative importance of the factors listed. At higher processing temperatures, errors in temperature control and timing become more important.

SUMMARY

1. Equations for the destruction of spores by heat are formulated and discussed.

2. The methods of process evaluation in common use are consistent with the definition of an adequate process as one which will reduce the "survival ratio" at the slowest heating point of the can to a prescribed value. By "survival ratio" is meant the ratio of the final to the initial concentration of spores at the point under consideration. It is suggested that an adequate process should be defined as one which will reduce the "survival number for the can" to a prescribed value. When the "survival number for the can" is less than 1.0, as it usually is, it may be interpreted as the probability of there being one surviving spore in the can at the end of the process.

3. It is shown that the new definition leads to estimates of process times required which are greater than those deduced by the older methods, the difference being larger for products heating by convection than those heating by conduction.

4. The relation between the consequences of the old and new definitions may be expressed by means of the concept of equivalent volume; the survival number for the can is given by the product of the equivalent volume and the survival ratio at the slowest heating point of the can. The equivalent volume varies with the nature of the product and also with the processing conditions. However it is shown that the variation with processing conditions is generally small compared with the inevitable uncertainties in some other data used in practical calculations. For products heating by convection the equivalent volume is approximately equal to the volume of the can, whereas in products heating by conduction it is about one-fortieth of the can volume.

5. Neglect of the cooling phase in evaluating processes is shown to be equivalent to an additional safety factor of less than 3 on the survival number scale for products heating by convection and a safety factor of the order of 10^7 in products heating by conduction in a No. 2½ size can.

6. It is shown that the point of minimum F value in a can of material heating by conduction is not necessarily at the center, but the F value at the center is generally very near the minimum value.

7. The relation between the formulae used by Ball and the exact temperature histories of products heating by conduction is discussed.

8. Some of the points which should be taken into account in a logical consideration of safety factors are discussed.

APPENDIX I. DERIVATION OF EQUATION (16).

We require the solution of the equation of conduction of heat in the finite cylinder $o \leq r < a$; $-L < x < L$; initial temperature T_1; surface temperature T_2, $o < t < t_1$ and T_3, $t > t_1$.

Write $T - T_1 = v$, $T_2 - T_1 = v_2$, $T_3 - T_1 = v_3$.
The equations to be satisfied are:

$$\frac{\partial v}{\partial t} = \alpha\left(\frac{\partial^2 v}{\partial r^2} + \frac{1}{r}\frac{\partial v}{\partial r} + \frac{\partial^2 v}{\partial x^2}\right), 0 \leq r < a, \quad -L < x < L, \tag{A1}$$

$$v = 0, \text{ when } t = 0, \tag{A2}$$

$$\left.\begin{aligned} v &= v_2,\ 0 < t < t_1, \\ &= v_3,\ t > t_1. \end{aligned}\right\} \text{ at } r = a \text{ and } x = \pm L. \tag{A3}$$

Put $v = u + w$,
where u and w each satisfy the equation of conduction (*A1*),

and $u = 0$ when $t = 0$, (*A4*)

$u = v_2$, $t > 0$, $r = a$ or $x = \pm L$, (*A5*)

$$\left.\begin{aligned} w &= 0, \quad t < t_1 \\ &= v_3 - v_2, \quad t > t_1 \end{aligned}\right\} \quad r = a \text{ or } x = \pm L, \qquad (A6)$$

As shown by Carslaw and Jaeger (*3, page 194*) the set of equations in u are satisfied by

$$u = v_2 \phi (r, x, t), \qquad (A7)$$

where the value of $\phi(r, x, t)$ is that already shown in equation (*15*), and βn, $n = 1, 2, \ldots$ are the positive roots of $J_0(\beta) = 0$. This is equation (*14*) of the text, and applies to the heating phase.
Similarly the set of equations in w is satisfied by:

$$w = 0, \quad t < t_1, \qquad (A8)$$
$$w = (v_3 - v_2)\, \phi (r, x, t - t_1). \quad t > t_1,$$

i.e. for $t > t_1$

$$v = u + w$$
$$= v_2 \phi (r, x, t) + (v_3 - v_2) \phi (r, x, t - t_1). \qquad (A9)$$

This is equation (*16*) of the text.

APPENDIX 2. RELATION BETWEEN EQUATION (16) AND THOSE USED BY BALL

For the heating phase Ball assumes that if $(T_2 - T)$ is plotted against time on semi-logarithmic paper, the later stages of heating, which are all we are concerned with, are well represented by a straight line. Fig. 1 shows that this is a valid assumption for the case considered. Analytically it means that all but the first term in each series in equation (*15*) is negligible.

If this straight line is extrapolated back to zero time it makes an intercept on the axis greater than $(T_2 - T_1)$. Ball defines the quantity j by writing:

$$\text{intercept} = j(T_2 - T_1).$$

From equation (*15*), for our case $j = \dfrac{8}{\pi} \dfrac{1}{\beta_1 j_1(\beta_1)}$

$$= 2.040$$

Experimental values of j for conduction heating packs are commonly less than 2.04. The reason for this is that in practice the temperature is not uniform in the can at the time selected as the zero of the time scale (often the time when the retort has been heated to T_2). Consequently experimentally determined j values and not the theoretical value of 2.04 should be used with Ball's formulae.

Ball represents the temperature difference between the retort and the centre of the can at the time steam is cut off, by g and the difference between retort temperature and cooling water temperature by $m + g$. In the present terminology g is the value of the first term in equation (*16*) when $t = t_1$.

The form of cooling curve used by Ball in preparing his Tables may be represented by plotting log $(T - T_3)$ against time (see Fig. 3). For large values of $(t - t_1)$ he assumed this gave a straight line of slope equal in magnitude but opposite in sign to that of the heating curve. From equation (*16*) it follows that this assumption is true for our case, for values of t large enough to make $\phi (r, z, t)$ and all but the first terms in the series in $\phi (r, z, t - t_1)$ negligible. Ball locates the linear part of the cooling curve on the diagram by assuming that when extrapolated back to $t = t_1$ it makes an intercept of 1.41 m. From equation (*16*) it follows that the intercept should be 2.04 $(m + g)$. Curve II in Fig. 3, derived from equation (*16*) shows that in experimental work, if the observations are ended at a temperature around 180°F., as is usually considered reasonable, it would be natural to deduce a value of the intercept close to that used by Ball, but a lower value for the slope of the cooling curve. Ball reports (*1a*) that cooling curves of this apparent form are commonly obtained.

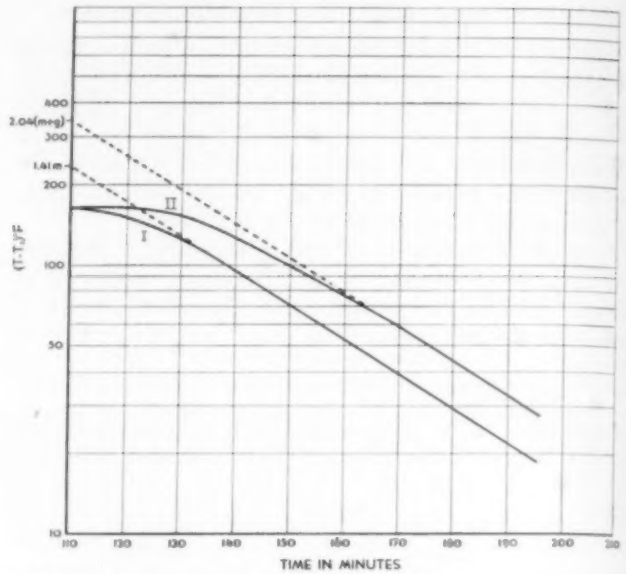

FIG. 3. Comparison of equation (*16*) with Ball's assumptions for cooling phase. Curve I. Ball's assumptions; Curve II. Equation (*16*).

Ball represents the part of the curve between the time steam is cut off and the point on the straight line at which $T - T_3 = 0.343m$ by a hyperbolic arc, i.e. by a monotonic decreasing function. The curved portion of curve II in Fig. 3 is of roughly the same form as Ball's hyperbola but it is not monotonic; it rises to a maximum at about 118 minutes. This cannot be shown very clearly on the scale of Fig. 3, but it is clearly seen in Fig. 1. A rise of a degree or so at this stage has a significant effect on the destruction of spores so that it should not be neglected.

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TABLE 1

Times at 250°F. (121.1°C.) necessary for specified spore populations in cans of various sizes.

Can size	Designation	Net contents liquid products, ml.	Minutes at 250°F. required to reduce spore population to 10^{-9} per can[a] from			
			10^9 per ml.	10^6 per ml.	10^3 per ml.	1 per ml.
No. 2	307 x 409	530	5.76	4.93	4.09	3.26
No. 2½	401 x 411	765	5.79	4.96	4.12	3.29
No. 10	603 x 700	2840	5.96	5.13	4.30	3.46
Tube	....	1	5.00	4.17	3.34	2.50

[a] The figure of 10^{-9} spores per can was chosen as a purely arbitrary end point.

TABLE 2

F values for the heating and cooling phases at positions in a No. 2½ size can of material of thermal diffusivity 0.000 cm.²/min. Retort temperature 240°F. (115.6°C.). Initial temperature at 180°F. (82.2°C.). 110 minute cook. Cooling water at 70°F. (21.1°C.).

Heating phase. F value in equivalent minutes at 250°F. (121°C.)

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	4.18	4.25	4.44	4.79	5.34	6.14	7.31
0.1	4.26	4.32	4.52	4.87	5.42	6.22	7.41
0.2	4.49	4.55	4.76	5.12	5.68	6.50	7.70
0.3	4.90	4.97	5.18	5.56	6.13	6.98	8.22
0.4	5.53	5.61	5.83	6.23	6.84	7.72	9.00
0.5	6.47	6.55	6.79	7.21	7.85	8.79	10.13
0.6	7.84	7.92	8.18	8.63	9.32	10.31	11.72

Cooling phase

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	2.87	2.83	2.70	2.46	2.07	1.60	1.09
0.1	2.78	2.75	2.64	2.41	2.05	1.60	1.10
0.2	2.55	2.53	2.45	2.29	2.00	1.60	1.10
0.3	2.20	2.20	2.16	2.07	1.88	1.55	1.11
0.4	1.80	1.80	1.80	1.76	1.69	1.46	1.10
0.5	1.36	1.36	1.39	1.40	1.38	1.28	1.05
0.6	0.97	0.98	0.99	0.99	1.01	1.02	0.91

Total F value—equivalent minutes at 250°F. (121.1°C.)

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	7.05	7.08	7.14	7.25	7.41	7.74	8.40
0.1	7.04	7.07	7.16	7.28	7.47	7.82	8.51
0.2	7.04	7.08	7.21	7.41	7.68	8.10	8.80
0.3	7.10	7.17	7.34	7.63	8.01	8.53	9.33
0.4	7.33	7.41	7.63	7.99	8.53	9.18	10.10
0.5	7.83	7.91	8.18	8.61	9.23	10.17	11.18
0.6	8.81	8.90	9.17	9.62	10.33	11.33	12.63

TABLE 3

F values for the same can as in Table 2 for a cook of 100 minutes.

Heating phase

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	2.80	2.86	3.02	3.30	3.76	4.44	5.47
0.1	2.86	2.92	3.08	3.37	3.83	4.52	5.55
0.2	3.05	3.11	3.28	3.58	4.05	4.75	5.81
0.3	3.39	3.45	3.63	3.94	4.44	5.18	6.27
0.4	3.92	3.99	4.18	4.52	5.05	5.83	6.98
0.5	4.73	4.80	5.01	5.38	5.95	6.78	8.01
0.6	5.93	6.01	6.24	6.64	7.27	8.17	9.47

Cooling phase

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	2.44	2.41	2.30	2.11	1.80	1.40	0.98
0.1	2.37	2.34	2.26	2.06	1.79	1.40	0.99
0.2	2.18	2.16	2.10	1.98	1.74	1.40	1.00
0.3	1.89	1.89	1.87	1.80	1.65	1.38	1.02
0.4	1.56	1.57	1.57	1.50	1.47	1.32	1.02
0.5	1.20	1.21	1.23	1.25	1.24	1.17	0.91
0.6	0.87	0.88	0.89	0.91	0.93	0.93	0.87

Total F value

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	5.24	5.27	5.32	5.41	5.56	5.84	6.45
0.1	5.23	5.26	5.34	5.43	5.62	5.92	6.54
0.2	5.23	5.27	5.38	5.56	5.79	6.15	6.81
0.3	5.28	5.34	5.50	5.74	6.09	6.56	7.29
0.4	5.48	5.56	5.75	6.02	6.52	7.15	8.00
0.5	5.93	6.01	6.24	6.63	7.19	7.95	8.92
0.6	6.70	6.79	7.13	7.55	8.20	9.10	10.34

TABLE 4

F values for the same can as in Tables 2 and 3 with a cook of 90 min.

Heating phase

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	1.74	1.78	1.91	2.13	2.49	3.04	3.89
0.1	1.79	1.83	1.95	2.18	2.54	3.10	3.96
0.2	1.93	1.97	2.11	2.34	2.72	3.30	4.19
0.3	2.20	2.24	2.38	2.64	3.04	3.65	4.59
0.4	2.62	2.67	2.82	3.10	3.54	4.20	5.20
0.5	3.28	3.33	3.51	3.82	4.30	5.03	6.11
0.6	4.29	4.35	4.55	4.91	5.46	6.26	7.45

Cooling phase

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	1.97	1.95	1.87	1.73	1.49	1.18	0.84
0.1	1.91	1.90	1.84	1.70	1.48	1.18	0.85
0.2	1.77	1.76	1.72	1.63	1.46	1.19	0.87
0.3	1.55	1.55	1.54	1.50	1.39	1.18	0.89
0.4	1.30	1.30	1.32	1.32	1.28	1.15	0.91
0.5	1.02	1.03	1.06	1.08	1.09	1.04	0.90
0.6	0.74	0.76	0.80	0.81	0.84	0.85	0.80

Total

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	3.71	3.73	3.78	3.86	3.98	4.22	4.74
0.1	3.70	3.73	3.79	3.88	4.02	4.28	4.81
0.2	3.70	3.73	3.83	3.97	4.18	4.49	5.06
0.3	3.75	3.79	3.92	4.14	4.43	4.83	5.48
0.4	3.92	3.97	4.14	4.42	4.82	5.35	6.11
0.5	4.30	4.36	4.57	4.90	5.39	6.07	7.01
0.6	5.03	5.11	5.35	5.72	6.29	7.12	8.25

TABLE 5

Reduction in spore concentration at positions in a No. 2½ size can of material of thermal diffusivity 0.090 cm.²/min. Retort temperature 240°F. (115.6°C.). Initial temperature 180°F. Cooling water at 70°F. (21.1°C.).

110 minutes cook. $\frac{C}{C_0} \times 10^{28}$

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	4.4	3.4	2.1	0.84	0.22	0.014	$<10^{-4}$
0.1	4.7	3.7	1.8	0.65	0.13	0.0074	$<10^{-4}$
0.2	4.7	3.4	1.2	0.22	0.024	$<10^{-3}$	$<10^{-5}$
0.3	2.9	1.6	0.40	0.036	0.0015	$<10^{-4}$	$<10^{-7}$
0.4	0.43	0.22	0.036	0.0018	$<10^{-4}$	$<10^{-7}$	$<10^{-10}$
0.5	0.068	0.035	$<10^{-3}$	$<10^{-6}$	$<10^{-7}$	$<10^{-10}$	$<10^{-14}$
0.6	$<10^{-5}$	$<10^{-6}$	$<10^{-7}$	$<10^{-8}$	$<10^{-11}$	$<10^{-14}$	$<10^{-19}$

100 minutes cook. $\frac{C}{C_0} \times 10^{13}$

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	1.42	1.11	0.73	0.35	0.100	0.0098	$<10^{-4}$
0.1	1.54	1.20	0.62	0.29	0.061	0.0065	$<10^{-4}$
0.2	1.54	1.11	0.44	0.10	0.015	$<10^{-3}$	$<10^{-5}$
0.3	1.02	0.62	0.16	0.023	0.0012	$<10^{-4}$	$<10^{-7}$
0.4	0.19	0.10	0.021	0.0022	$<10^{-4}$	$<10^{-6}$	$<10^{-9}$
0.5	0.0047	0.0024	$<10^{-3}$	$<10^{-4}$	$<10^{-6}$	$<10^{-9}$	$<10^{-12}$
0.6	$<10^{-5}$	$<10^{-5}$	$<10^{-6}$	$<10^{-5}$	$<10^{-10}$	$<10^{-13}$	$<10^{-16}$

90 minutes cook. $\frac{C}{C_0} \times 10^{14}$

r/a	x/L						
	0.0	0.1	0.2	0.3	0.4	0.5	0.6
0.0	4.5	3.8	2.5	1.3	0.48	0.066	$<10^{-4}$
0.1	4.9	3.8	2.4	1.1	0.35	0.040	$<10^{-4}$
0.2	4.9	3.8	1.7	0.52	0.092	0.0071	$<10^{-4}$
0.3	3.2	2.3	0.79	0.13	0.012	$<10^{-3}$	$<10^{-6}$
0.4	0.79	0.52	0.13	0.013	$<10^{-3}$	$<10^{-5}$	$<10^{-7}$
0.5	0.034	0.021	0.0059	$<10^{-3}$	$<10^{-5}$	$<10^{-7}$	$<10^{-11}$
0.6	$<10^{-4}$	$<10^{-4}$	$<10^{-5}$	$<10^{-6}$	$<10^{-9}$	$<10^{-11}$	$<10^{-15}$

TABLE 6

Approximate effects of some factors on estimates of the chance of survival of a spore in a No. 2½ size can processed at 240°F. (115.6°C.).

Factor	Estimate of n_f multiplied by	
	Convection heating	Conduction heating
Neglect of equivalent volume effects	500	14
Neglect of cooling phase	1/3 to 1	10^5
Temperature 1°F. below nominal value	3.5	3.5
Length of cook 1 min. less than nominal	20	4

Problems in Chocolate Liquor Production[a]

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A REVIEW

Some standardization in the chocolate industry is necessary to understand the terms used when referring to cacao trees, beans, and the different products obtained in processing. The food technologist has little or no control over the fermenting of cacao beans as primitive methods are used in cacao producing areas. The Inter-American Cacao Center is trying to set up standards for the different methods of producing good, uniform, high quality cacao beans. Roasting is the most important process in bringing out the flavor in chocolate and the continuous roasters transferring heat by convection and conduction produce the finest roasted cacao beans. Advantages of American made Aluminum Oxide Triple Mill Stones over Natural French Burr Stones is discussed and means of maintaining high production of nibs to liquor and liquor to liquor are shown, together with a method for the fast redressing of Aluminum Oxide Stones by diamond-impregnated wheels.

Cacao (Ka-cow or cä-cäó), chocolate liquor, and cocoa are not synonymous terms. To avoid confusion a standardization is necessary. The writer, in line with the majority of scientists throughout the country working in the interests of the industry, prefers cacao to describe the tree and the bean. After roasting and processing in the factory, the bean is freed of its shell and germ to produce cocoa nibs which are ground to make chocolate liquor. Cocoa is the pulverized cake that remains when part of the cocoa butter is expressed from the chocolate liquor. This cocoa powder is used in the preparation of various beverages, cake and fudge mixes. The chocolate standards of the Federal Security Agency define chocolate coating as a product made from chocolate liquor derived from cocoa nibs.

Cacao is one of the plants that was in cultivation in the Americas at the time of their discovery. It was in the Matina Valley of Costa Rica where cacao under cultivation was first found by Christopher Columbus in 1501 and it is still one of the best cacao areas in the world today. Cacao was always highly regarded by early Indian civilizations and was reserved for the use of the ruling class. It is a natural food concentrate, a source of quick energy, and a body-builder as well. More than one-half of it is edible fats, and the balance contains sugars, starches, and proteins, plus a small amount of a harmless stimulant. It is nature's K ration, ready-packed, and was used extensively by the military forces of this country. It has been used as a unit of money and was indeed a good money as it was scarce, could be stored or hoarded and was naturally formed into small units, but, most important, it has intrinsic value as a food. At some time a good slave could be purchased for ten cacao beans *(5)*.

[a] Presented at the Tenth Annual Meeting of the I. F. T., Chicago, Illinois, May 24, 1950.

The raw cacao bean itself is the tart, rather unpalatable, acrid seed of the cacao tree and must be fermented and roasted to bring out the characteristic flavor and aroma that has made chocolate the top flavor in the food industry. Unfortunately food technologists have little or no control over the fermenting of the cacao bean. This step of fermenting and drying of the cacao beans, which are grown in tropical climates of South America, Africa, and many of the neighboring islands that lie in the equatorial belt, is performed by primitive methods by relatively primitive people and it is a miracle that any fermentation takes place at all or that there is an uniformity or control. These farmers usually dig a hole in the ground, open the pods and place the beans in this hole and cover them up with leaves where they are left to ferment. This action kills the germs and produces many changes in the cacao bean. Needless to say it is important that the heat of fermentation be controlled for a given length of time, after which these beans are washed of their fermentation juices, sun-dried, and put into bags. The proper drying is a problem as the climate in these tropical countries changes rapidly and many moldy beans result and they are passed on by exporters to countries of commerce.

THE INTER-AMERICAN CACAO-CENTER

There has been organized an Inter-American Cacao Center *(8)* whose objectives are as follows: (1) to discover, devise, and evaluate methods which can be used to produce a greater quantity of cacao of higher quality at a lower unit cost; (2) to train students to use these methods properly, adapt them intelligently to local conditions, conduct similar research in their own countries, and introduce the results of research effectively into the cacao producing industry; and (3) to disseminate the information gained as widely and as rapidly as possible.

This Inter-American Cacao Center has been doing very good work and it is heartening to know that better cacao beans are on the way. The fermentation process tentatively recommended by the Center *(6)* is as follows:

1. The beans are extracted from the pod and placed in boxes for fermentation. The boxes are covered with several thicknesses of heavy burlap, to retain the heat of fermentation. The contents of each box are turned every 12 hours until the fourth day, when the fermentation is complete.

2. The mass of fermented pulp and beans is then placed in a revolving perforated drum placed in a tank. Water is admitted to the tank and the beans are then dumped into perforated dump wagons and allowed to drain.

3. The washed and dried beans are then partially dried on trays in a hot-air dryer. They are then placed in a segmented drum and tumbled. Hot air is passed through the drum during the tumbling, and this completes the drying. In addition to the drying, the tumbling action polishes and hardens the beans and results in a more desirable product both in appearance and in shipping quality. It will take years before these standards are generally adopted.

The purchasing and inspection of cacao beans as they arrive at the factory is a continuous problem and constant checking must be maintained. The various types of cacaos have their own distinct flavor and aroma and proper blending of different types is essential.

The next important step is the cleaning of the beans in special equipment which will remove strings, sticks, stones and any other foreign materials. Sometimes fumigation is necessary.

THE ROASTING OF CACAO

The food technologist has little to do with fermenting, but he should watch the roasting operation which can be controlled. Roasting is the most important step in the production of proper flavor in chocolate.

Many manufacturers use batch roasters in which there is very little control. During a day's run some batches are under-roasted or over-roasted and some obtain very little roasting at all. Such batch processes as a rule produce some beans that have black, partly burnt shells that do not add to the flavor of the beans. Radiant heat is not very effective in producing proper roasting of cacao beans. The intensity of radiant heat varies inversely as the square of the distance from the source; therefore, the matter of distribution is very critical. Cacao beans that are further away from the radiant surface are roasted slowly and come out under-roasted while beans that are nearer are burnt or over-roasted. Another difficulty is that heat absorption increases as the bean grows darker in roasting. Even the raw beans vary in color which makes it almost impossible for a batch of beans to absorb heat uniformly. Therefore, radiant heat roasting is difficult and expensive and the quality of the product is not held uniform.

The transfer of heat *(4)* by convection and conduction has been improved by the construction of continuous roasters which automatically control the velocity of the heat medium from a constant source of heat. The temperature of the hot air that surrounds the cacao beans is kept uniform within narrow limits. By recirculating the air it is possible to attain fuel economy in the continuous type roasters. Continuous roasters produce a product of uniform and controlled roasting at low temperatures, give better heat transfer, and require little or no skill in operation. They are economical and deliver a superior product of the proper flavor and aroma which is so necessary in the production of good chocolate.

After the beans are roasted, they are taken to winnowing devices known as cracking and fanning units in which the beans are cracked open and the nibs, the meat of the cacao beans, separated from the shell and germ. This process is very important and must be carefully inspected so that the percentage of shell and germs will be below the Federal Standard of 1.75 percent. Today continuous cracking and fanning units are available that take the discharge of a continuous roaster and automatically do this job very efficiently.

These nibs are the starting point in the manufacturing of chocolate. They pass through the various types of grinding machinery in which they are reduced to a creamy, smooth paste, the product we all know as chocolate liquor.

GRINDING OF CACAO

The reduction of nibs to chocolate liquor has always been a problem in the chocolate industry. The nibs contain over 50 percent of a fat which has a low melting point and for this reason they do not lend themselves to pulverization. They also contain about 3 percent of fiber which adds to the difficulty when it comes to re-

ducing nibs to fine particle sizes. The first commercial machine used for the reduction from nibs to liquor is still commonly used in most manufacturing concerns today. This machine is known as a "Triple Burr Mill" which is equipped with "Natural French Burr Stones" that consist of three separate sets of round stones, each set having different size "bosoms," furrows, and feathering designs. The "Natural French Burr Stones" have a short life between dressings, are not uniform as they have hard and soft spots, and glaze easily. A considerable amount of contamination results in their wearing down. Also as the stones wear, the pounds per hour productivity drops rapidly and they have to be redressed quite often in order to obtain good production of chocolate liquor.

The art of sharpening of these stones has always been a closely guarded secret by a certain few stone cutters who supposedly are the only ones who have the know-how for this type of work. The redressing operation is a ritual in itself. Certain tools must be used, certain chisels must be tempered just so, certain weight of hammers used and the redressing operation is a slow, laborious and costly procedure. The chocolate liquor obtained with these Natural French Stones must usually undergo further reduction in particle sizes in various types of refining machines before the chocolate liquor can be used for making a high grade sweet chocolate.

Today, through American ingenuity and know-how, modern plants are employing a ceramically bonded, crystalline, aluminum oxide stone with controlled grain sizes instead of the natural stones (Figure 1). These

FIG. 1. Bottom of a 30-inch standard mill stone.

Aloxite stones *(3)* can be readily made in a latitude of grain sizes ranging from very coarse to very fine. These stones are more uniform and therefore they have no hard or soft spots. They have good strength and excel in thermal shock. They will wear evenly and will outlast natural stones and give higher initial and continuous production as well as a better product. Redressing periods are cut by 80 percent.

Aloxite stones are sharpened first with resinous bonded grinding wheels which revolve at high speeds and regroove the feathering designs that were worn down. The resinous wheels are very thin and break very easily as they have to follow a curved groove. Another problem is that they wear down very rapidly in the redressing of Aloxite stones and constant adjustment must be made. We took up this problem with the manufacturer of the Aloxite stones and had them make two diamond-impregnated wheels, six inches in diameter, 0.045 inches in thickness and with 5/8 inch core. One we will call F and the other with 50 percent more diamonds H. We found that these diamond wheels would cut satisfactorily when dry and would feed down about 0.104 inch per pass. Using plain water as a coolant increased the cutting action by 50 percent, and practically no adjustments were needed for the regrooving operation on the Aloxite stones (Figure 2).

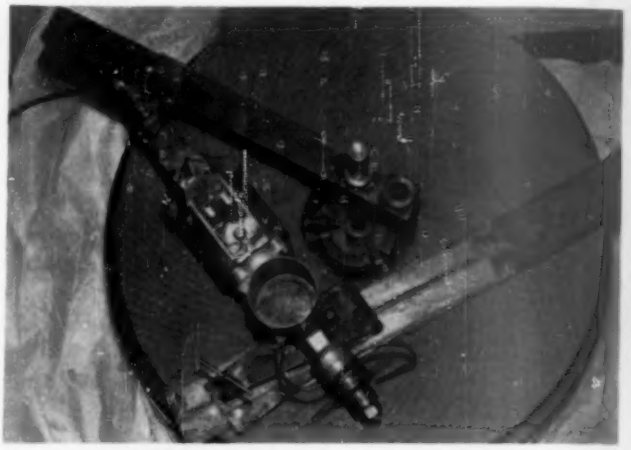

FIG. 2. Diamond wheel in position to resharpen or for redressing feathering designs of standard mill stone.

Diamond wheels for feathering mill stones will cut more freely and have more life at a high peripheral speed. In fact, the recommended maximum is 12,000 surface feet per minute; thus with the 6 inch diameter diamond wheel the maximum r.p.m. should be 7,700. We ran our tests using a 3800 r.p.m. grinder using plain water as a coolant and the time for feathering one 36 inch stone was about 6 hours, there being 160 grooves to each Aloxite stone. This same operation for resinous bond cut-off wheel would take about 18 hours per stone, and 20 resinous wheels. Other disadvantages of the resinous type wheel are their flexibility and easy breaking and the feathering is very poor. The life of the F wheels with the lower concentration of diamonds was about one-half of the H wheels with more diamonds. The price difference of that size diamond wheel is approximately $5.00. These tests were run on a machine designed in our own machine shop, using one diamond wheel and one pass in resharpening the worn grooves.

By using a "Triple Mill Stone" with the top stone having a grit of 80 and the second set a grit of 150 and the bottom stone a grit of 280, it is possible to obtain a good quality chocolate liquor. With the use of the proper "bosoms," furrows, and feathering designs, and the passing of the chocolate liquor from one mill to another with the second mill employing fine grit size, it is possible to obtain chocolate liquor at a rate that will

surpass any other type equipment available on the market.

The capacity of liquor mills in grinding nibs to liquor is as follows: On a 48 inch diameter mill: 1000 lbs. per hour; on a 36 inch mill: 600 lbs. per hour; and on a 30 inch mill: 400 lbs. per hour. The capacity of grinding coarse liquor to fine liquor on a 48 inch mill will reach 1300 lbs. per hour; on the 36 inch mill: 1000 lbs. per hour; and on the 30 inch mill: 600 lbs. per hour. Thus, it is apparent that in an hour 600 lbs. of nibs can pass through a 36 inch mill and be pumped to a 30 inch mill, this resulting in 600 lbs. of very fine chocolate liquor ready to be used in chocolate coatings without any further processing. To increase production, cacao nibs can be pre-ground in a Mikro-Pulverizer *(7)* which must be equipped with a screen, that has $\frac{1}{16}$ inch diameter round openings. Thus one can take advantage of the liquor to liquor capacities of Aloxite mill stones.

From this chocolate liquor, unlimited blends of chocolate confections can be derived, depending upon the particular blend of cacao beans employed, the percentages and types of sugar, cocoa butter, milk solids, and flavors combined therewith, the fineness to which the ingredients are milled, adjustment to various viscosities, and percentages of chocolate liquor used. Well-known examples are: sweet chocolate, which by Federal Standards must contain at least 15 percent by weight of chocolate liquor; bittersweet chocolate, which must contain at least 35 percent by weight of chocolate liquor; and milk chocolate, which must contain at least 10 percent by weight of chocolate liquor.

The manufacture of chocolate coatings is generally conducted in accordance with certain time-honored customs and procedures and by the use of certain generally accepted standardized types of machinery such as melangeurs, refiners, and conches. Most of this equipment is characterized by the consumption of excessive time and labor.

The complete pulverization of cocoa nibs has been tried many times but has proven unsuccessful as far as obtaining a product that does not need further particle size reduction. The closest approach to nibs pulverization has been disclosed in a recent patent *(1)* in which freshly and properly roasted nibs are cooled and used immediately in a complete chocolate formula, adding materials such as sugar, milk powder, and salt as scourging agents. These materials are put through a Mikro-Atomizer mill which has been cooled and maintained below the melting temperature of the cocoa butter. A complete one-step process of making chocolate is thus possible. This process has an advantage to manufacturers who have no initial investment in the Aloxite Triple Mills.

Greater production of chocolate is possible with the separate grinding of cocoa nibs to chocolate liquor by "Triple Mill Stones," and used in conventional processes or the one step method *(2)* where the dry ingredients (sugar, milk powder, etc.) are reduced together to the correct particle size and added to the chocolate liquor, and cocoa butter, and other liquid ingredients.

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Detection of Deterioration Products of Autoxidizing Milk Fat by Infrared Spectrophotometry[a,b]

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The course of autoxidation of milk fat was studied by changes in the infrared absorption spectra of the volatile components, in the acceptability of the fat, and in its peroxide value. Spectral changes were detectable before a trained panel of observers could detect flavor changes. Flavor changes were marked before peroxide value changed appreciably. Loss of flavor is correlated with a specific absorption band, and growth of off-flavors with several others. The nature of the deterioration products formed is discussed.

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[b] This paper reports research undertaken at the Quartermaster Food and Container Institute for the Armed Forces, and has been assigned number 307 in the series of papers approved for publication. The views or conclusions contained in this report are those of the authors. They are not to be construed as necessarily reflecting the views or indorsement of the Department of Defense.

In the course of a study of the deterioration during storage of dried milk products, it had been observed that taste panels composed of trained observers frequently rejected as unacceptable products which had none of the usual chemical criteria of deterioration. Since a part of the reason for rejection was described as off-flavor believed to be due to lipid deterioration, a more

thorough investigation of the autoxidation of milk fat was indicated. Storage studies of commercial dry milk fat also demonstrated that rejection due to off-flavor and odor occurs before such chemical criteria as peroxide value indicate deterioration.

Infrared examination of autoxidizing milk fat suggested itself as a possible means for detecting products of deterioration before they had accumulated in quantities sufficient for chemical estimation. Preliminary investigations indicated that the quantities of the components necessary to produce considerable off-flavor and odor are too small to be readily detectable, even in the infrared absorption spectrum, when still dissolved in the milk fat. However, sharp absorption peaks due to hydroperoxide have been observed at 2.9 μ in samples with relatively low peroxide value (PV = 2), but with distinctly off-odor. A similar peak in autoxidizing linseed oil has been reported by Daubert *(3)*.

Sharp changes in the carbonyl absorption region (6 μ) are not discernible in the whole fat, because of the prominence of the ester carbonyl absorption at 5.7 μ. The presence of carbonyl compounds was suspected, however, as they have been detected in other autoxidizing fats *(2, 5)*. It was further suspected that these carbonyl compounds should be volatile under proper conditions, since steam deodorization is an established technique for other malodorous fats; e.g., Daubert recovered hexenals from the distillate of reverted soybean oil *(2)*. Accordingly, a sample of malodorous milk fat was distilled with steam *in vacuo*, and the distillate examined by infrared absorption spectrometry. Carbon disulfide proved to be a suitable solvent for the extraction of the organic distillate. The residue from the extract was examined in the infrared, and several bands were found in the 6-μ region, especially at 5.77, 5.81, 5.91, and 6.11. These bands constituted evidence of the presence of at least one unsaturated, conjugated ketone (or aldehyde) and another which was not conjugated. Aldehyde appears to be eliminated because the distillate did not give a positive test with Schiff's reagent.

An experiment was devised to follow the development of these new substances in milk fat and to correlate them, if possible, with the taste acceptance of a milk in which the fat is recombined. A batch of fresh fat was divided, and the control portion stored at —18° C. in sealed jars. The experimental aliquots were stored in open beakers in an air oven at 45° C. Test samples were removed semi-weekly from both storage and control. In addition to peroxide value and infrared study, milks of 3.5 percent fat were made up by recombining, using a method similar to that of Krukovsky *(4)*, the storage and control fats with a standard non-fat milk solids and distilled water. The resulting milk was submitted to sensory difference testing.

Peroxide value remained essentially constant during the first 3 weeks of the test (Figure 1), and then rose, gradually at first, and after 4 weeks more sharply. Sensory discrimination data, by duo-trio test *(6)*, show a somewhat increasing awareness of deterioration during the first two weeks, which is without statistical significance. Between the 16th and 21st day

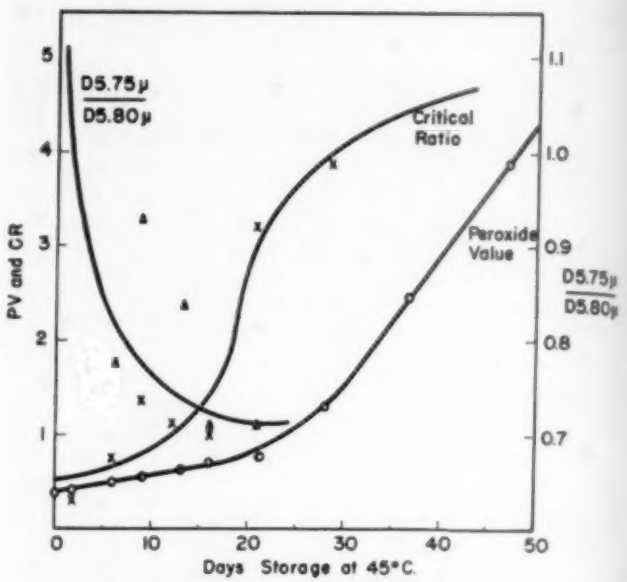

FIG. 1. Peroxide value (PV), sensory difference data (CR), and infrared criteria for milk fat stored in air at 45° C.

the deterioration, as indicated by taste, became statistically significant, and by the 28th day had reached the level where further testing was not necessary. This is shown as the Critical Ratio (CR) in Figure 1. Subjective descriptions of the fat were changed from "buttery" to "flat" during the 1st week, then to "strong" and finally to "rancid" during the 3rd week.

During this 28-day period, the infrared spectra of the whole stored fats showed no discernible changes. But the spectra of the steam-volatile components were most interesting. Only the 3-μ and the 6-μ regions have been examined in detail, since it is in these regions that most significant changes are to be expected.

Sampling for these spectra was as follows: The distillate from steam-deodorization at 95° C. of the stored fat was condensed in a dry-ice trap. Deodorization of a 400-g. sample required 3 hours and consumed 300 ml. water. After thawing, the distillate was extracted twice with 50-ml. portions of carbon disulfide, the extract dried, and its volume reduced to about 1 ml. by evaporation. This residue of the extract was then plated dropwise on the sample cell window, the solvent evaporated, and spectra run on a Perkin-Elmer 12-C infrared spectrometer. Since sample thickness in this method is unknown, comparison between samples is extremely difficult. The following arbitrary expedient was adopted to avoid much of this difficulty. Blanks were run on the same chart as the sample with a thick salt window in the path. Optical densities (OD) were determined by point-to-point subtraction between blank and sample on the Perkin-Elmer logarithmic paper. The peak of the greatest density for each region of interest was then set arbitrarily at OD = 1, and the densities at all other points factored appropriately. The resulting factored densities were then plotted on single-cycle semi-logarithmic paper so that samples could be compared visually. Hydroxyl absorption at 3.1 μ increases in intensity and shifts slightly to a longer wave length, indicating increasing association.

The changes in absorption in the 6-μ region of these samples are shown in Figure 2. In the distillate from the fresh fat only two peaks are observed, one at 5.80 μ, ascribed to diacetyl by Rasmussen *(7)*, and one at 5.75 μ which has not yet been assigned. Upon storage,

the peak at 5.80 μ remains quite constant, while that at 5.75 either disappears or shifts to 5.70. At 45° C. this occurs within the first 6 days, while at —18° C. it is only partially complete after 4 months. It is during this period that the flavor changes from "buttery" to "flat." It is upon this evidence that the presence of a sharp peak at 5.75 μ can be used as indication of freshness in the fat, and one at 5.70 μ as an indication of flavor loss.

Upon longer storage, new peaks appear at 5.84 and at 5.91 μ. The former is probably due to a simple ketone *(7)*. The latter and a smaller peak at about 6.10 μ become increasingly strong with time, and are ascribed to a conjugated enone or enal. These bands are shifted about 0.1 μ each from those expected for isolated C = O and C = C respectively *(7)* Although there is insufficient spectral evidence to permit differentiation between ketone and aldehyde, ketone is preferred because these distillates did not give the color test with Schiff's reagent. This ketone is believed to be associated with the off-flavor which develops in the fat on storage since similar substances have been reported found in autoxidizing methyl linoleate *(1, 2, 4)*. To date no mechanism for its formation has been offered.

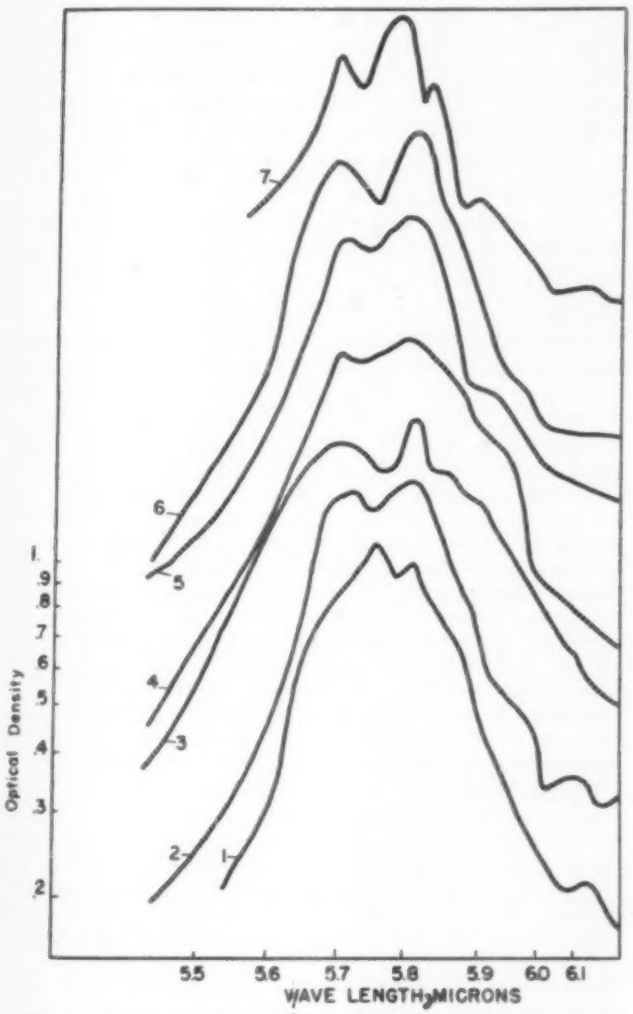

FIG. 2. Infrared absorption spectra in the carbonyl region of distillates from milk fat (each line vertically displaced 0.2 O. D. units). 1. Fresh. 2. Stored 4 months at —18° C. 3-7. Stored at 45° C. 6, 9, 13, 16, and 21 days, respectively.

DISCUSSION

Since spectral evidence for a ketone is present before the end of the first week of storage at 45° C., while sensory discrimination appears significant only after two weeks, it is suggested that the shape of the infrared absorption curve of the steam-vacuum distillate of milk fat in the region 5.5 to 6.20 μ is a more sensitive test of freshness than is a trained taste panel. The latter appears to be more sensitive in detecting deterioration than peroxide values. It is further suggested that a quantitative method may be developed whereby the flavor and acceptability of fats and food products containing fats can be predicted from infrared absorption measurements, and perhaps, storage life estimated. This would in large measure reduce the time required for determination of acceptance, and eliminate much of the element of guess work still present in panel testing.

SUMMARY

Infrared absorption studies of the steam volatile components of autoxidizing milk fat have indicated that this method can detect changes before they are apparent by chemical or sensory means. The absorption maxima of these components show that they contain one or more simple ketones (or aldehydes), and a conjugated ketone.

ACKNOWLEDGMENT

The cooperation of Mr. D. Peryam and the Food Acceptance Branch of this Institute in conducting and evaluating the duo-trio test for acceptability is gratefully acknowledged.

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Preparation of a Frozen Apple Juice Concentrate[a]

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Three methods for the preparation of frozen apple juice concentrate have been investigated and the products compared organoleptically. Data are included which indicate that the concentration can be carried out in relatively low-cost vacuum pans without affecting the flavor of the product.

The commercial success of frozen orange juice concentrate has created a great deal of interest in the commercial possibilities of concentrated juices prepared from other fruits. This interest has led to several questions with regard to selection of the optimum conditions for manufacture and storage of such concentrates. This paper reports part of an investigation designed to furnish the desired information for frozen apple juice concentrate. In particular, it deals with the temperature range best suited to concentration of the apple juice and with evaluation of methods of furnishing natural flavor to the concentrate. Results show that apple juice may be concentrated at temperatures considerably higher than those used commercially for the concentration of orange juice. Hence, commercial vacuum pans, used in conjunction with essence recovery units, appear to be suitable for the preparation of frozen apple juice concentrate. Full-flavored concentrates have been prepared by three different methods, each of which appears to be technically suitable for commercial use, and storage studies are under way on both canned and frozen samples.

As practically all of the volatile flavoring constituents in apple juice are lost during vacuum concentration, investigators have attempted either to remove water from juice by freezing or to recover the volatile flavoring constituents during vacuum concentration. In the former case, it has been assumed that volatile constituents remain with the concentrate during ice removal, while in the latter it is intended that these constituents be later incorporated in the concentrate. Considerable work has been done on "freeze" concentration *(5, 6, 12, 16)*, but no practical equipment has yet proved entirely satisfactory for commercial use, although one type is now being considered seriously *(1)*.

Several methods for preparation of concentrates with full fresh apple flavor have been proposed. Carpenter and Smith *(3)* suggested use of an essence impregnator *(14)* by means of which the first fraction of distillate during concentration is combined with previously concentrated apple juice. Poore *(13)* outlined a method in which the first fraction of distillate (9 percent) was collected during vacuum concentration of apple juice. This fraction was concentrated separately by redistillation and then added back to the main batch of concentrate. Mottern *(11)* suggested a system in which a major portion of the vapors was condensed, while the remainder was collected in a cold trap and its contents later thawed and added to the concentrated juice.

A commercially successful method for recovery of natural apple flavors from apple juice was described by Milleville and Eskew *(9, 10)* and a public service patent issued to Milleville *(8)*. Juice was heated to boiling temperature and approximately 10 percent by weight vaporized in a tubular heat exchanger. The vapor was led through a fractionating column and the flavoring constituents obtained in an aqueous solution whose volume was 1 percent (or less) of that of the original juice. Times of exposure in the hot zones for both juice and essence were of very short duration; hence heat damage to either material was negligible. Among various uses suggested by the authors was incorporation of this product in a full-flavored concentrate in the manner reported by Poore *(13)*.

Preparation of frozen apple juice concentrate by a method similar to that commercially used in the preparation of frozen orange juice concentrate has been investigated by the Maine Experiment Station *(2)* and Cruess and Gibson *(4)*. Several frozen apple juice concentrates are now being test marketed *(1)*.

METHODS

Juice Preparation. Combination Fancy and Extra Fancy grade apples were purchased on the local market for use in these experiments. A blend of 70 percent Winesap and 30 percent Delicious was fed into a stainless-steel hammer mill and pressed in a small rack-and-frame press. The juice was depectinized by addition of 0.1 percent by weight of a commercial enzyme preparation for pectin degradation and allowing the mixture to stand overnight at 50° F. (10° C.). Clarification was accomplished by filter aid added to the juice and passing the mixture through a small plate filter which had been precoated with the same material. The clarified product from a group of pressings was combined, placed in 30-lb. cans,[c] frozen, and stored at —10° F. (—23° C.) in order to provide a uniform stock of juice for each phase of the experimental program.

Essence Stripping. Volatile flavor constituents were separated by partial volatilization of the apple juice, and were concentrated by rectification of the vaporized fraction. One method for carrying out the process has been described by Milleville and Eskew *(9, 10)*. In the work described here the juice was preheated to 212-217° F. (100-102.8° C.) in about 1 second by the direct injection of steam, and 18 to 20 percent of the juice was vaporized in about 2 seconds during a single pass through a short tubular evaporator following the preheater. The equipment used will be further described elsewhere by M. E. Lazar and T. Wasserman. The vapors containing the volatile flavors were concentrated in a fractionating column to 1 percent of the volume of the original juice. The residue from the stripping operation (stripped juice) was later concentrated to the desired

[a] Presented before the Tenth Annual Meeting of the I. F. T., Chicago, Illinois, May 21-25, 1950.

[b] Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Report of a study made under the Research and Marketing Act of 1946.

[c] All cans used in these experiments were coated with fruit enamel.

degree, and combined with the volatile flavor concentrate, in order to form a full-flavored concentrate.

Concentration. Concentration of both fresh and stripped juice was carried out in a laboratory-size falling-film evaporator *(15)*. The unit consists essentially of a vertical, jacketed evaporating tube operating on the principle of a falling-film, a vapor-liquid separator, a condenser, a series of cold traps immersed in an alcohol-dry ice mixture, and an arrangement for continuous recirculation of juice during concentration. Its operating range is 20 to 300 mm. absolute pressure and it is so arranged that practically all of the products of evaporation are retained in the system. Pressure was held at a constant figure for the duration of each run with the result that the temperature within the system increased slowly as the juice became more concentrated. Consequently, in discussions concerning temperature, nominal temperatures of evaporation (defined as the boiling points of water at the pressures employed) are used. All references to the degree of concentration attained are given on a volume basis. Depectinized juices were used, since six-fold concentrates prepared from pectin-containing juices gelled rapidly after removal from the evaporator and the gel dissolved with difficulty during reconstitution with water.

Sample Preparation. Samples for organoleptic evaluation were prepared by two methods. When changes brought about by concentration in the falling-film evaporator were to be evaluated, concentrate, distillate, and material caught in the traps (recovered by washing out with 200 ml. of distillate) were combined, sealed in 307 x 409 cans, frozen, and stored at —10° F. (—23° C.) until needed for appraisal. At the time of appraisal, the samples were thawed by placing the cans in running water; the cans were opened and the contents adjusted to 70° F. (21° C.) before tasting.

When samples were prepared for evaluation of various types of full-flavored concentrates, the completed four-fold concentrates were packed in concentrated form. Those for frozen storage were placed in 211 x 212 cans, sealed, frozen, and stored at 0° F. (—18° C.). Those for room-temperature storage were heated to 200° F. (93° C.) in a high-velocity pasteurizing tube, hot-filled into 211 x 212 cans, sealed, inverted for 3 minutes, cooled in running cold water, and stored at 75° F. (24° C.). When needed for appraisal, the samples were opened, diluted with exactly 3 volumes of distilled water, and adjusted to 70° F. Samples of single-strength juice were packed for comparison with the full-flavored concentrates. This juice was obtained from the same lot used in preparing the concentrates and was packed in 307 x 409 cans by the same methods used in packing the concentrates.

Organoleptic Evaluation. Evaluations of samples were made by a panel of 15 experienced judges. When two samples of juice were to be evaluated, judging was done by the triangulation method (six replications) as described by Helm and Troll *(7)*. When more than two samples of juice were to be evaluated, they were judged as a group, with sufficient replications so that results could be analyzed statistically. Samples of the latter type were scored on an arbitrary scale of values from 10 to 1 on the basis of (a) fullness of natural flavor and (b) freedom from off-flavor. To prevent color differences from influencing the judges, all tastings were conducted in a room lighted solely by a sodium vapor lamp.

EXPERIMENTAL RESULTS

Effect of Low-Temperature Concentration Upon Apple Juice. The effect of concentration was investigated in order to establish the effectiveness of the evaporator unit in retaining volatile constituents within the system and to find whether significant changes took place in any of the constituents in the juice during evaporation. A portion of juice was concentrated to six-fold at an absolute pressure of 31 mm. (see Table 1 for temperatures), the fractions were recombined, and the product was compared with the remainder of the juice.

The taste panel found no significant differences between the two types of juice, in either aroma or flavor, although the juice which had been concentrated was appreciably lighter in color. Hence, it appears that the loss of volatile flavor constituents from the evaporation unit is negligible and that no chemical changes affecting flavor take place during evaporation under these conditions.

Effects of Various Temperatures of Concentration. An important factor in the choice of a commercial evaporating unit is the maximum temperature at which evaporation can be carried out without appreciably affecting the flavor of the product. To determine this maximum permissible temperature for apple juice, samples of six-fold concentrates were prepared in the laboratory unit at evaporating temperatures of 85°, 100°, 115°, 130°, and 145° F. (29°, 38°, 46°, 54° and 63° C.). The evaporating period for each run was approximately 3 hours. Operating data consisting of nominal temperatures employed and corresponding absolute pressures, boiling temperatures of juice at the beginning and end of concentration, and temperatures used in the jacket surrounding the falling film column are listed in Table 1.

TABLE 1

Operating Data Obtained During Concentration of Apple Juice

Run No.	Absolute pressure (mm.)	Nominal b.p. of water (° F.)	Boiling point of juice (° F.)		Jacket temperature (° F.)
			Start	Finish	
1	31	85	87	92	150
2	49	100	102	107	160
3	76	115	117	123	175
4	115	130	133	139	185
5	170	145	148	154	200

The results show that concentration can be carried out at temperatures up to 130° F. (2.22 p.s.i.a., 115 mm.) without significant change in flavor of product, provided the concentration unit is designed so that localized overheating of juice does not occur. Comparison of recombined juices prepared at 85°, 100°, 115°, 130°, and 145° F. also showed that (a) samples prepared at 145° F. were significantly different in flavor from those prepared at lower temperatures and (b) the color became progressively lighter with increase in temperature of concentration.

Comparisons of Various Types of Full-Flavored Concentrates. Samples of full-flavored apple-juice concentrate were prepared by three methods for organoleptic evaluation in order to determine (a) differences in flavor due to method of preparation, (b) effects of storage at room temperature (canned) and at 0° F. (—18° C.) (frozen) for a period of a year upon these differences, and (c) keeping qualities of these concentrates as compared with those of single-strength juice.

The methods of concentrate preparation, each of which was considered to be feasible for commercial use, were as follows:

A. Juice was concentrated to six-fold and the concentrate cut back to four-fold with fresh juice.

B. Juice was stripped, the residue concentrated to slightly over four-fold, and an equivalent amount of concentrated volatile flavor constituents added to the concentrate.

C. Juice was stripped, the residue concentrated to six-fold, an equivalent amount of concentrated volatile flavor constituents added to the concentrate, and the blend cut back to four-fold with fresh juice.

For this set of experiments, concentration was carried out at 125° F. (52° C.). Each lot of ful-flavored concentrate was divided into two equal portions, one of which was packed for frozen storage and the other for room-temperature storage. Samples of the single-strength juice used in preparing these concentrates were packed in a similar manner.

Differences in flavor, ascribable to the method of concentrate preparation, were found when reconstituted samples were compared nine days after the packs had been completed. A summary of the results obtained from comparison of frozen and canned samples of the concentrates and single-strength juice is presented in Table 2. The scores on "fullness of natural flavor"

TABLE 2

Organoleptic Evaluation of Single-Strength and Concentrated Apple Juice

Type of juice	Frozen [d]		Canned [e]	
	Freedom from off-flavor [f]	Fullness of natural flavor [g]	Freedom from off-flavor	Fullness of natural flavor
Single strength	7.8	7.6	6.8 [h]	7.0
4-fold concentrates:				
Method A (6X + fresh juice)	8.4	5.9 [h]	8.4	6.3 [h]
Method B (4X + essence)	8.5	7.9	8.7	8.1
Method C (6X + fresh juice + essence)	8.1	7.8	8.5	7.7

[d] Stored 9 days at 0° F. [e] Stored 9 days at 75° F. [f] Score of 10: no off-flavor. [g] Score of 10: full natural flavor. [h] Indicates significant differences ($P = 0.05$).

received by samples of both frozen and canned concentrate prepared by Method A were significantly lower than those received by the other samples. These results indicate that concentrates prepared by Method A are perceptibly deficient in natural flavor as compared with the original juice, while concentrates prepared by either Method B or Method C appear to have about as much natural flavor as the original juice. A significant difference in the "freedom from off-flavor" was found only in the canned single-strength juice.

After three month's storage of the frozen concentrates, no perceptible changes in off-flavor rating or fullness of natural flavor were apparent. Further results of the storage studies will be reported later.

Effect of Pasteurization. Flavor changes occurring during pasteurization were investigated. Samples of concentrate packed for room temperature storage (prepared by Method B) were reconstituted and compared organoleptically with those packed for frozen storage within 48 hours after preparation. It appears that no change in flavor was caused by pasteurization, per se, for the panel found no significant difference between the canned and the frozen samples.

Vacuum Pan Concentration. One lot of four-fold full-flavored concentrate was prepared on a pilot-plant scale. Seventy gallons of clarified juice consisting of an 80-20 blend of Winesap and Delicious apples was processed by Method B. The same equipment was used for stripping as that used in the preparation of the previous concentrates. Concentration was carried out in a vacuum pan equipped with an agitator. The jacket temperature was maintained at 190° F. and the pressure at 1.5 p.s.i.a. (78 mm.). The concentrate obtained from this run was equal to any prepared in the laboratory concentrator.

DISCUSSION OF RESULTS

Full-flavored concentrates of excellent quality can be prepared from fresh apple juice. Concentrates prepared by stripping juice prior to concentration and adding back the volatile flavor constituents to the concentrated juice (Method B, a four-fold concentrate plus essence, and Method C, a six-fold concentrate plus single-strength juice plus essence) were found to be superior in flavor and aroma to those prepared by the method similar to that currently in use for commercial preparation of frozen orange juice concentrate (Method A). No significant differences in either flavor or aroma were found between samples prepared by Method B and those prepared by Method C. Either of these two methods appears to be equally suitable for commercial use, and the choice between them seems to depend largely on economic considerations. Storage-stability differences that may show up later in the storage study could result in a somewhat different evaluation of the three methods.

SUMMARY OF RESULTS

1. The material obtained by concentration of juice to six-fold at a nominal temperature of 85° F. (29° C.) and recombination of the products of concentration was indistinguishable in flavor from that of the original juice.

2. Recombination of the products of concentration resulted in a juice which was considerably lighter in color than the original juice. The change in color became greater as the temperature of concentration was raised.

3. Evaporation of juice at temperatures up to 130° F. (54° C.) did not affect the flavor of the recombined product. However, use of a temperature of 145° F. (63° C.) resulted in a product containing a distinguishable amount of off-flavor.

4. Four-fold concentrates containing volatile flavor constituents obtained by stripping of juice before concentration were considered superior in flavor to six-fold concentrates made four-fold with single-strength juice.

5. In concentrates which contained recovered volatile flavor constituents, no significant differences in flavor were found between samples in which four-fold concentrate served as a base and those in which six-fold concentrate had been cut back with single-strength juice.

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The Effect of Leavening and Shortening Combinations on the Frozen Storage of Cakes and Cake Batters Prepared at High Altitude[a]

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Baked cakes and cake batters prepared from formulae adapted to an altitude of 5000 ft. were held at —10° F. (—23.5° C.) from one to four months. Formula variations included two types of baking powder (sodium-aluminum-sulphate and tartrate) and three kinds of shortening (hydrogenated vegetable oil, butter, and lard). Tests on freshly baked cakes, thawed frozen cakes, and cakes made from batters frozen in the baking pan and in cartons included measurements on specific volume, compressibility, moisture, palatability, and pH.

Previous studies on the freezing of cakes and cake batters have given little information on the use of a formula adapted to a high altitude. Reports in the literature *(4, 6, 7, 8)* indicate that freezing may cause a decrease in the volume of baked cakes and of cakes baked from frozen batters at lower elevations. Modifying the amount of baking powder and other ingredients in a recipe to compensate for the decrease in atmospheric pressure *(2)* might affect the volume still further, particularly in the cakes baked from frozen stored batters since these are subjected to more handling and storage in the liquid stage. The type of baking powder used could also influence the quality of the finished product.

Recommendations concerning the best form for freezing cakes and batters are not in agreement. Graul and Lowe *(4)* have reported that frozen baked cakes made with either hydrogenated lard or butter as the shortening had better palatability than similarly formulated cakes made from frozen batters. Miller and Beattie *(7)*, and Owen and Van Duyne *(8)*, using hydrogenated vegetable oil as the shortening found that the frozen batters gave a more desirable baked product than the frozen cakes. Since different types of shortening were used in the above studies perhaps the type of shortening may have influenced the quality of the product. The method of thawing the frozen cakes may also have influenced the quality of the product. Although both Graul and Lowe *(4)* and Miller and Beattie *(7)* thawed the frozen cakes at room temperature, Owen and Van Duyne *(8)* heated the frozen cakes to an internal temperature of 122° F. (50° C.) before judging.

The present investigation was designed to study the effect of frozen storage on the quality of cakes and cake batters made from a plain, shortened, cake formula adapted to the altitude at which the study was conducted (5000 ft.). Variations in the formula included two types of baking powder and three types of fat. Baked cakes, and cake batters in baking pans and in cartons were frozen for periods of one to four months. Under normal conditions of home freezing, cakes are not held in frozen storage for longer periods.

EXPERIMENTAL METHODS

Preparation of Cakes and Batters. The ingredients for a plain, shortened, cake formula developed for use in this laboratory (5000 ft. elevation) were as follows:

Cake flour	302.0 g.
Sugar	410.0 g.
Shortening:	
Hydrogenated vegetable oil, OR	140.0 g.
Butter OR	140.0 g.
Lard	140.0 g.

[a] Contribution from Montana State College, Agricultural Experiment Station Paper No. 235, Journal Series.

Salt	8.4 g.
Baking powder:	
Sodium-aluminum-sulphate type OR	10.6 g.
Tartrate type	22.4 g.
Mixed whole egg (frozen)	220.0 g.
Homogenized milk	333.0 ml.
Vanilla	7.0 ml.

In the remainder of the paper hydrogenated vegetable oil will be referred to as HVO, sodium-aluminum-sulphate baking powder as SAS, and tartrate baking powder as Tartrate.

In order to assure uniform ingredients throughout the study, the cake flour, sugar, shortenings, and baking powders were obtained from single lots and in ample quantity to complete the study. Sufficient whole egg was mixed and frozen in amounts needed for four formulae *(5)*.

The batter was prepared by a modification of the "quick-mix" method described by Redfield *(9)* using a home-model electric mixer. All procedures were standardized. For the fresh cakes, the frozen cakes, and frozen pans of batter, 275 grams of the mix were weighed into six-inch round aluminum cake pans lined with waxed paper and lightly greased. Waxed cartons (capacity one pint) were filled with 365 grams of batter for freezing. The pans of batter to be tested as fresh cakes and frozen cakes were baked at 350° F. (176.5° C.) for 33 minutes prior to freezing while the others were frozen as batter and baked after storage. Cakes and batters were weighed before and after frozen storage.

Each batch was sufficient for four six-inch cakes and there were five replications of each treatment. The respective portions were distributed at random among the various treatments as indicated in Table 1. Work on each treatment for the four

TABLE 1

Variations in Formula, Form of Frozen Product, and Months of Frozen Storage [b]

Formula Variations	Form of Frozen Product			Months of Frozen Storage				
	Baked Cakes	Pans of Batter	Cartons of Batter	0	1	2	3	4
SAS-HVO	+	+	+	+	+	+	+	+
Tartrate-HVO	O	+	+	+	+	+	+	+
SAS-Butter	+	O	+	+	O	+	O	+
SAS-Lard	+	O	+	+	O	+	O	+

[b] + indicates combinations included in study; O indicates those omitted.

formula variations could not be included; however, it will be noted that the experiment was designed to give comparative information on each of the variations studied.

Freezing. After cooling one hour the freshly baked cakes were wrapped in waxed paper using a drug-store wrap, placed in two-quart moisture-vapor-proof freezer bags and heat-sealed. The pans of batter were heat-sealed directly in the freezer bags, excluding as much air as possible. The cakes and pans of batter were then placed in lightweight cardboard boxes to prevent crushing during storage. The baked cakes, and the pans and cartons of cake batters were stored in a commercial home freezer at —10° F. (—23.5° C.) for one to four months.

Thawing. At the end of each storage period certain of the frozen baked cakes were removed from the boxes, thawed in the freezer bags for 15 minutes at 300° F. (149° C.), unwrapped, and allowed to cool *(3)*. The frozen batters were thawed at room temperature prior to baking—the pans of batter (in the freezer bags) for 30 minutes; the cartons for one hour and 45 minutes. Two hundred and seventy-five grams of the batter which had been stored in cartons was baked in pans at 350° F. (176.5° C.) for 37 minutes.

Testing. The cakes were allowed to cool for three hours after baking or thawing before volume, weight, compressibility, moisture, and palatability measurements were made. In order to express numerically the lightness of the cakes, the volume/weight relationship (specific volume) was determined. Volume measurements were made using the rape seed displacement method. Four one-inch cubes were cut from the center of each cake with the aid of a miter box and compressibility determinations were made on the samples using a Baker Compressimeter with a stress of 320 grams. Moisture analyses were made on all cakes following the air-oven method as given in Cereal Laboratory Methods *(1)*. pH determinations were obtained for freshly mixed batters and thawed frozen batters.

At the end of each storage period samples of the cooled, baked products were coded, placed on a tray together with a fresh control sample and rated by qualified judges. The rating sheet devised for this study was based on a maximum score of 100 points with four points assigned to each of the five external characteristics: shape, volume, crust color, crust surface, and crust texture. Grain was given 18 points; crumb texture, 14; moistness, 7; and flavor, 41 points, since the latter factor was considered most important.

Analysis of Results. The data obtained on specific volume, compressibility, and moisture content were treated statistically according to the method of analysis of variance *(10)*. "F" values were determined and differences in means necessary for significance at the one percent and five percent levels were calculated to determine when significant changes occurred.

RESULTS AND DISCUSSION

Mean specific volumes of the fresh cakes, frozen stored cakes, and cakes made from the frozen batters ranged from 3.08 to 2.51 (Table 2). Fresh cakes made

TABLE 2

Mean Specific Volume [c] *of Fresh and Thawed Frozen Cakes and Cakes from Frozen Batters*

Formula Variations	Form of Frozen Product	Months of Frozen Storage					D. M.[d]	
		0	1	2	3	4	5% Level	1% Level
SAS-HVO	Cake	3.08	2.96**	2.97	2.94	2.93	0.08	0.12
	Pan	3.08	2.98	2.99	2.92**	2.90	0.11	0.15
	Carton	3.08	3.08	3.02	3.00	3.00	.....	.....
Tartrate-HVO	Pan	3.02	2.76**	2.66	2.58	2.62	0.17	0.23
	Carton	3.02	2.79**	2.70	2.71	2.75	0.08	0.12
SAS-Butter	Cake	2.75	.....	2.64	.....	2.76	.....	.....
	Carton	2.75	.....	2.62*	.....	2.63	0.11	0.15
SAS-Lard	Cake	2.60	.....	2.57	.....	2.51	.....	.....
	Carton	2.60	.....	2.52	.....	2.51	.....	.....

[c] Specific volume = volume (ml.) ÷ weight (g.).
[d] Difference in means necessary for significance.
**Significant at the 1 percent level.
*Significant at the 5 percent level.

with HVO and either SAS or Tartrate had higher specific volumes, 3.08 and 3.02, than did those with SAS and either butter or lard, 2.75 and 2.60 respectively. Frozen storage caused a significant decrease in the specific volume of baked cakes and cakes prepared from pans of batter with SAS and HVO; of the cakes from batters frozen in the pan or in the carton with Tartrate and HVO; and of the cakes made from SAS and butter batters frozen in the carton. The specific volumes of the cakes with SAS and lard were not affected by frozen storage. The decreases noted were observed in most cases at the time of the first test period, indicating that the changes had occurred early in the storage period. Following the initial decrease the specific volumes of some products decreased slightly with longer storage, though most changes beyond the first test period were not significant. Owen and Van Duyne *(8)*, studying the effect of frozen storage on cake volumes, report similar findings.

When either the SAS-HVO or Tartrate-HVO variation was used, cakes made from the cartons of frozen batter had higher specific volumes than those from pans of frozen batter. This latter finding is also

supported in the study conducted by Owen and Van Duyne *(8)*. The baked products using SAS-butter and SAS-lard had much lower specific volumes than those made with SAS-HVO indicating that the type of fat used affected the specific volume of the products.

Mean compressibility values of the fresh cakes and frozen stored cakes and of cakes made from the frozen batters are given in Table 3. There were no significant

TABLE 3

Mean Compressibility of Fresh and Thawed Frozen Cakes and Cakes from Frozen Batters [e]

Formula Variations	Form of Frozen Product	Months of Frozen Storage					D. M.[f]	
		0	1	2	3	4	5% Level	1% Level
		mm.	mm.	mm.	mm.	mm.		
SAS-HVO	Cake	1.50	1.63	1.66	1.56	1.61	.....	.....
	Pan	1.50	1.33	1.35	1.35	1.22	.....	.....
	Carton	1.50	1.42	1.32	1.46	1.36	.....	.....
Tartrate-HVO	Pan	1.65	1.37	1.11**	0.98	1.04	0.36	0.50
	Carton	1.65	1.48	1.33*	1.26	1.38	0.25	0.35
SAS-Butter	Cake	1.98	.....	1.68	.....	1.96	.....	.....
	Carton	1.98	.....	1.81	.....	1.88	.....	.....
SAS-Lard	Cake	1.32	.....	1.21	.....	1.40	.....	.....
	Carton	1.32	.....	1.31	.....	1.21	.....	.....

[e] Compressibilities were made using a Baker Compressimeter and a stress of 320 grams.
[f] Difference in means necessary for significance.
**Significant at the 1 percent level.
*Significant at the 5 percent level.

changes during frozen storage in the compressibility of any of the products made with SAS baking powder. However, the cakes made with Tartrate were decidedly less compressible after two months of frozen storage. At the end of four months' storage the frozen baked cakes with SAS-HVO, or SAS-butter, or SAS-lard were more compressible than the cakes made from the respective types of frozen batter.

There were no significant changes in the weight of any of the products during storage. The mean moisture content of the experimental cakes is given in Table 4. The moisture content of the SAS-HVO and SAS-lard cakes decreased significantly after one and two months of storage, respectively, although longer storage periods did not result in further decreases. The products made with Tartrate-HVO and SAS-butter were not appreciably lower in moisture content. In most instances the cakes baked before freezing contained less moisture than did those from the frozen batters. When the frozen cakes were thawed, drops of moisture were frequently observed on the inside of the wax-paper wrapping and the crusts were soft and gummy; this observation has also been made by other workers *(3, 4)*.

In general the average pH of the SAS batters was from 6.2 to 7.1 and the tartrate batters from 6.2 to 7.2. The pH values of the frozen batters did not differ appreciably from those of the fresh batters and hence are not presented. This finding agrees with that of Meyer, Buckley, and Moore *(6)*.

The mean palatability scores are presented in Table 5. After three or four months of storage the SAS-HVO frozen, baked cakes and cakes from the frozen batters compared favorably with the fresh control samples. However, a few undesirable compact layers occurred at the bottom of these cakes. This phenomenon has been

TABLE 4

Mean Moisture Content of Fresh and Thawed Frozen Cakes and Cakes from Frozen Batters

Formula Variations	Form of Frozen Product	Months of Frozen Storage					D. M.[g]	
		0	1	2	3	4	5% Level	1% Level
		%	%	%	%	%		
SAS-HVO	Cake	23.44	21.59**	21.87	20.89	20.91	1.34	1.84
	Pan	23.44	22.15**	22.59	23.54	22.01	0.72	0.99
	Carton	23.44	22.18*	22.14	23.51	22.90	1.06	1.46
Tartrate-HVO	Pan	22.86	23.12	22.93	23.05	22.64	.....	.....
	Carton	22.86	22.94	22.59	23.27	23.80	.....	.....
SAS-Butter	Cake	24.92	.....	23.69	.....	23.97	.....	.....
	Carton	24.92	.....	24.45	.....	24.45	.....	.....
SAS-Lard	Cake	23.91	.....	22.55**	.....	22.07	0.78	1.08
	Carton	23.91	.....	22.76*	.....	22.95	0.85	1.17

[g] Difference in means necessary for significance.
**Significant at the 1 percent level.
*Significant at the 5 percent level.

TABLE 5

Mean Palatability Scores [h] *for Fresh and Thawed Frozen Cakes and Cakes from Frozen Batters*

Formula Variations	Form of Frozen Product	Months of Frozen Storage				
		0	1	2	3	4
SAS-HVO	Cake	97 (0) [i]	93 (0)	96 (0)	96 (0)	91 (3)
	Pan	97 (0)	96 (0)	98 (0)	95 (0)	94 (1)
	Carton	97 (0)	94 (0)	96 (1)	94 (0)	94 (2)
Tartrate-HVO	Pan	96 (0)	90 (3)	89 (4)	82 (4)	89 (4)
	Carton	96 (0)	93 (2)	94 (3)	96 (1)	88 (5)
SAS-Butter	Cake	96 (0)	.....	90 (4)	.....	90 (5)
	Carton	96 (0)	.....	93 (2)	.....	86 (4)
SAS-Lard	Cake	62 (0)	.....	69 (2)	.....	57 (5)
	Carton	62 (0)	.....	66 (2)	.....	57 (4)

[h] Perfect score = 100.
[i] The figures in parentheses indicate the occurrence of compact layers among the five replications.

noted by other workers *(4, 7, 8)*. When Tartrate-HVO was used, the cakes baked from pans or cartons of frozen batter had a compact layer after only one month of storage. At the end of three and four months the palatability of these cakes was considerably less than that of the fresh product and practically all of the cakes had this undesirable, heavy layer. These results, together with the findings on specific volume and compressibility of the cakes made with the Tartrate-HVO variation, indicate that the tartrate baking powder was not as satisfactory as the SAS type for the freezing of cake batters when the fat used was hydrogenated vegetable oil. When the SAS-butter and SAS-lard variations were used this compact layer was present in most products after four months of storage, indicating that the cakes made from the butter and lard with the SAS baking powder were not as desirable as those made with the SAS-HVO variation. This finding is supported by the mean specific volumes of the cakes given in Table 2 since specific volume is a measure of the lightness of the cake. All products made with lard had an objectionable lardlike flavor which was not affected by storage.

No off-flavors developed in any of the products during the four months of storage. Meyer, Buckley, and Moore *(6)* report off-flavor development after six months' storage. The crusts of the thawed frozen baked cakes were soft and gummy and the crumb was somewhat drier than that of cakes made from frozen batters; these characteristics have been observed by other

workers *(3, 4)*. It is of interest to note that, when significant changes took place in the experimental cakes (Tables 2, 3, and 4), the change was evident early in the storage period.

SUMMARY

Quality measurements of the cakes made using the formula variations SAS-HVO, Tartrate-HVO, SAS-butter and SAS-lard indicate that the cake formula including SAS-HVO was satisfactory for frozen storage at an altitude of 5000 feet. After four months of storage the cakes made from these batters, frozen in pans or in cartons, were similar in quality to the freshly baked control samples. Cakes made from the batters frozen in the carton for four months were slightly superior to those frozen in pans.

The formula with Tartrate-HVO was not satisfactory for frozen storage under the experimental conditions of this study. Specific volume, compressibility and palatability values were lowered after storage and most products had an undesirable compact layer.

The formulae with SAS-butter or SAS-lard were not satisfactory for frozen storage. The undesirable heavy layer was present in most products after storage and specific volumes of these cakes were considerably less than those made using SAS-HVO. Quality ratings of the lard cakes were particularly low.

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Recent Advances in Coffee Technology

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The recent developments in coffee growing, in roasting and in the soluble coffee industry are reviewed. A summary is given of some recent developments in fundamental research in coffee flavor.

At first glance a cup of coffee seems pretty trivial. It appears nowhere in the Government nutrition charts. However, more American families regularly use coffee than any other table served item except possibly salt and sugar. Over 90 percent of all families serve coffee. Over 80 percent of all individuals over 16 years of age drink coffee, as do 15 percent of the kids over 6. Last year nearly 46,000,000,000 cups of coffee were consumed in the U. S. A.

Per capita consumption was 12.1 lbs. in 1929; about 14.4 lbs. prewar and about 20 lbs. in 1949-1950. The value of coffee imports is now by far larger than that of any other imported product, exceeding one-half billion dollars. Coffee has played an important part in the social and political life of England and Europe. "Coffee houses" were for years the center of literary and social activity in England. Charles II tried to suppress them as centers of political agitation.

In an effort to highlight some of the changes that have happened in the centuries old coffee industry over the last few decades and at the same time not skip anything of importance, these developments have been broken down into four fields of activity:

1. Developments occurring in the coffee growing areas of the world.
2. Developments taking place in the coffee roasting industry.
3. Developments taking place in the soluble coffee industry.
4. Fundamental Research.

GREEN COFFEE DEVELOPMENTS

Brazil supplies about one-half of the world's coffee. The 1950 Brazilian coffee crop is estimated to have been about 15 million bags. Carryovers will not exceed 5 million bags. Commercial agreements already negotiated will guarantee shipment of 6 million bags. Coastwise and port consumption will absorb 2 million bags. This leaves a net of 12 million bags or less for the United States from Brazil. The U. S. will probably use a total of 19,000,000 or about 65 to 75 percent of the world exportable production.

Interests in plantings have been stimulated and in 1951 and 1952 it is estimated that 250 million trees in Sao Paulo; 200 million in Parana will start producing which means an increase of 4 million bags each year.

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It is interesting to note that the production of coffee in Brazil has ranged from approximately 12 million bags in the period from 1900 to 1905; increased to about 15 million bags in the period from 1905 to 1920; remained at about 15 million bags from 1920 to 1925; and between 1925 and 1935 ran as high as 29,600,000 bags. After that there was a tendency to reduce and the period 1935-1940 dropped back to 20 million and to about 15 million bags in 1945. The period 1945-1950 would average something under 15 million bags.

The same information is presented in still more startling form when we attempt to combine this data and the total number of trees in such a way as to get yield per tree:

Year	Pounds/Tree
1920	1.30
1925	1.22
1930	1.22
1935	1.10
1940	.98
1942	.80
1944	.53
1946	.80
1948	.96
1950	.90

While there are many interesting observations that could be drawn from these data, the one in particular that needs to be emphasized at this time is the downward trend of the yield in the last 15 years. The situation in Brazil might be said to be somewhat akin to what occurred in our Southern states in the heavy cotton growing areas. The deep, rich soils of the tropical and semi-tropical areas of Brazil where coffee was originally grown, permitted a tree to bear for periods said to be as long as 75 years. However, no serious program of fertilization was followed during the twenties' and as a result, like our Southern cotton soils, these very rich coffee soils have been slowly exhausted. The industry, therefore, has found it necessary to migrate. This migration, in the main, has been toward the South to lighter soils and areas that are more susceptible to frost. The net result is that today the normal coffee-bearing life of a tree is about 15 to 20 years and even this period does not have the steady reliability of the old plantings because of the potential frost hazard; meaning a shorter productive life span and a reduced yield for each coffee tree. This shift in planting is illustrated by the following comparisons:

Area	1900	1920	1930	1940	1950
Sao Paulo	8,932	10,246	19,490	10,234	7,340
Minas Gerais	3,137	4,202	5,135	3,223	2,726
Rio de Janeiro	1,264	1,144	1,115	505	450
Espirito Santo	.....	484	1,579	1,160	1,805
Parana	.....	111	596	1,108	1,663

As a result of reduction in yield per tree and soil depletion, scientific fertilization by means of chemicals on an extensive scale, is now being tried in Brazil. It is much too early to comment on the success of this development, but it is something we should all watch with interest. In Central America, Colombia and Venezuela, there has been a marked increase in the use of mulches. Again in Brazil, insect control is moving to the forefront and dusting and spraying with benza-hexachloride has been practiced on a fairly broad scale.

Another trend which is taking place is an effort to upgrade and improve the quality of the coffee being produced. Small experimental demonstrations of carefully hand-picked and hand-cured Santos beans have shown there was possible an obvious improvement in character due to a better job of grading and eliminating of under-ripe and over-ripe beans. Some work is going on with control fermentation leading toward the same objective.

Some in the coffee industry believe that the differences recognized in the designations "Rio," "Santos" and "Central American," and other "type" coffees are perhaps much more attributable to methods of handling than has heretofore been recognized. Effort is being directed and will undoubtedly continue to so process Brazilian coffees as to give them Milds or Central American character. Even the coffee growers themselves are recognizing some of this trend, as evidenced by the installation of mechanical dryers, which provide for better control and reduce the damage due to rain during the drying period. These dryers are expensive, however, and require some mechanical understanding. As a result, progress has not been as great to date as it is expected to be in the future. Even in those areas where dryers are not being considered, there is a general move in the direction of improved drying patios and better methods of handling and protecting the cherries during this drying period. Along with this development of improved drying facilities, modern machinery for washing coffee is being installed and greater care is being taken during the fermentation step.

In brief, the coffee grower is slowly but surely recognizing the fact that his curing operation is based upon scientific principles and that the application of these principles even at the farm or fazenda will provide him with an improved item which will be more acceptable to his customers.

ROASTED COFFEE DEVELOPMENTS

As far as the technical side of roasted coffee is concerned, the industry normally thinks that this operation breaks down into three stages: a. Processing; b. Control; and c. Packaging.

Processing. From the standpoint of processing, not a tremendous amount of advancement has been made except in terms of size of the operation.

Coffee can be blended to any taste that one wants. It can be roasted to any desired shade: it can be ground pretty much as one pleases—to a coarse grind, a fine powder or pulverized grind or in between. In fact, all these processes are so simple that you can approximate them in your own kitchen. Fifty years ago the processing of coffee was mostly carried out in small corner local roasting and distributing operations or by small local wholesalers operating one or two roasters and packing under their own private brands. There is still an awful lot of this kind of business carried on today.

There are almost 1,000 roasters in the United States who roast over 1,000 brands, yet half the total coffee sold in grocery stores is in 5 brands.

The emergence of nationally distributed brands has resulted in increasing the scale on which operations are carried out, but in the main the blending of different types of beans to produce a uniform blend is still left to the individual judgment of expert coffee tasters in much the same way as the wine industry has operated for so many years.

Most roasters operate with identical machines which are practically unchanged since Jabez Burns invented the first roaster in 1881. A small roaster may use one machine. One of the larger roasters may use better than 50 machines operating 24 hours a day.

In the last few years new equipment has appeared and at the present time continuous roasters are in use in many of the large scale processing plants. These roasters are said to have the

advantage of greater uniformity than batch roasters and to contribute less shrinkage or loss of weight during the roasting operation.

More recently, a patent issued to a Dr. Wm. A. Lenz and covering a closed cycle roaster has been offered to the trade, which is said to have many advantages including freedom from release of odorous gases to the atmosphere, improvements in yield and improvements in quality. This particular development is so new and recent that it has not actually had what one might call a "college try."

Control. Probably one of the most far-reaching improvements in the coffee roasting business has been brought about by the application of controls of one form or another to the coffee roasting operation. In the past a so-called standard color or standard roast was established by the expert coffee taster and it was then the goal or objective of the roaster operator to visually match this sample as nearly as possible. In a large plant, where many people were operating roasters, individual interpretations of color were necessary, and it is easy to see how variations in shade of roast occurred.

This picture becomes still further complicated when we learn that the color of a standard would change with age, moisture content and several other factors. There has, therefore, been a concerted effort on the part of larger manufacturers of coffee to provide an objective measurement of this factor. Efforts have been made to provide mechanical control of the roast by temperature, but to date this has not proved feasible since different blends require different temperatures before arriving at a satisfactory roast condition. In most instances, therefore, the problem has been solved successfully by the application of photocolorimetric techniques and the correlation of sample measurement with previously established non-changing standards. In general, where these procedures have been applied, they have resulted in a marked tightening of the roast control operation and a definitiveness that has not permitted drifting from pre-established standards. The net result to the consumer has been uniformity of product over long periods of time. This is probably the most outstanding advancement in the processing of coffee in the last few years.

Another development which has come about quite recently has been the application of war-evolved electronic temperature controls to roasters, which is said to provide a mechanized means for duplicating the product from roast to roast, even in batch operations. In this system the mean temperature is taken through a very sensitive thermocouple which is intimately in contact with the beans in the roaster. At the precise end point, which has been correlated with color, a contact in the instrument causes automatic shutdown of firing and opening of the damper. At the same time a water spray is initiated for a timed period, which effectively retards further temperature development of the beans. At the end of the timed period a signal flashes and the roast is dumped by hand, or even this can be included in the automatic cycle. A gain of one full roast per hour's operation is claimed.

Another feature about which many of us are not aware is the shift of blend preferences which has taken place in the last few years.

There are two general classifications of coffee—Brazils and Milds. Up to a few years ago, the bulk of the coffee consumed in the United States was Santos, the Brazils. The Milds, largely grown in Central America and Colombia were too expensive for widely sold blends. The first extensive users of Milds coffees were West Coast roasters. They began to use Milds back in the early thirties because the ports of Colombia and Central America were more accessible to them than those of Brazil. Thus the cheaper transport minimized the cost differential. It is interesting to note that as people got used to these mild coffees over a period of time, increasing numbers liked them better than Santos. Wherever predominantly Santos blends were in competition with Milds, the Santos blends lost ground. This has resulted in a marked change in character of coffee during the last ten years. The change has been slow and progressive and has moved from the West Coast to the East over a period of several years. It has been necessary to make the changes in this manner because there were originally not enough mild coffees. As demand and plantings increased, production was stepped up and with increased production, practically all coffee manufacturers have slowly modified their formulas to incorporate more Milds in line with the changing taste of the American people.

In this particular case, the coffee manufacturer has introduced a more expensive ingredient into his formula because he felt that a superior product would result even though this superiority was not immediately recognized by the customer.

Packaging. Much has been said and much research has been carried on having to do with the proper packaging of coffee and the protection of coffee after it is roasted and before it is consumed. Probably the most far-reaching development in this regard was the introduction of vacuum packed coffee a number of years ago, since when the packers have constantly striven for increasingly high vacuums. At the present time, all of the high quality coffees on the market are being packed under vacuums of 29 inch or better, and experiments are being carried on at even higher vacuums. The net result has been a marked reduction or elimination of many of the old staling complaints which were so common to the industry. Work, however, in this field is still active as indicated by the following quotation from one paper by Punnett whose work was largely supported by the American Can Company:

1. The swelling of coffee during roasting is probably due to the internal production of gases, the main constituent being carbon dioxide, in tiny cells or pores in the beans. Measurements indicate that the pressure of these gases may range from 225 to 350 p.s.i. when the coffee is in the roaster. After cooling the gas pressure is of the order of 110 to 170 p.s.i.

2. The grinding of roasted coffee breaks open many of these cells and part of the gas is lost. The balance escapes through the cell walls into the container. If vacuum packed the gas may just about fill the empty space. If packing is done quickly after grinding there may be enough to produce some pressure eventually. A longer interval between grinding and packing may result in loss of enough gas so that the container will finally have a slight vacuum.

3. Roasted coffee is very susceptible to attack by the oxygen of the air. Measurements show that oxidation is fairly rapid during the first 20 days. During the next 6 months the reaction

occurs at a much slower rate but it speeds up enormously during the succeeding 8 to 10 months. It finally slackens but is still going on after almost 6 years.

4. A study of the process suggests that flavoring substances are concerned during the first three weeks (loss of coffee flavor and formation of staleness); a slow combination of oxygen with the oil in the coffee during the second period; a rapid reaction of oxygen with the oil in the third period with the production of true rancidity; and finally a much slower production of the oil and possibly other substances.

5. If the above is an approximately accurate picture of the deterioration of roasted coffee that is exposed to the air, then it is inaccurate to talk about "rancidity" or "rancid products" in coffee unless the exposure has lasted for at least 7 or 8 months.

Another series of papers by Elder and Shuman reports that the staling of coffee could not be correlated with the oxidation of the fatty oil fraction, but could at least be associated with the loss of carbon dioxide and the presence of oxygen. In this particular experiment the conclusion was drawn that the development of stale flavor in roasted coffee becomes progressively greater as the following pack variations are made:

1. Hermetic vacuum pack.
2. Ventilation with moist or dry nitrogen.
3. Hermetic pack with added moisture, no oxygen.
4. Hermetic pack with small amounts of dry oxygen.
5. Ventilation with dry oxygen.
6. Ventilation with moist oxygen.
7. Hermetic pack with small amounts of oxygen and added water.
8. Hermetic pack with oxygen at atmospheric pressure.

Work in the same laboratories has indicated the greatest change taking place during staling is the conversion of diacetyl to acetyl methyl carbinol reducing the diacetyl content from 10 to 5 p.p.m.

Still more recently, an effort has been made to correlate much of the information in the literature with freshness testing. This work has been done at General Foods Corporation and has not heretofore been reported. It might be summarized as follows:

Regular grind coffee was packed under a series of vacuum levels, 29.6, 27, 25, 20, and 15 inches, and taste-tested for freshness at intervals over a storage period of eighteen months. Results showed that all the oxygen in the headspace had disappeared after one month in the 29.6, 27, and 25-inch vacuum packs. In the same period, 80 percent of the oxygen sealed in the 20-inch vacuum pack and 70 percent of that in the 15-inch pack had disappeared. After three months, no oxygen remained in any of the cans. Taste test scores remained substantially unchanged after one to two months storage, and indicated recognizable stale flavor only in coffee packed at 20 inches or lower vacuum. Loss of grinder fresh quality was suspected even in the highest vacuum pack after about seven months, but taste scores were inadequate to prove this conclusively.

The method of brewing coffee beverage has an important bearing on the ability of a discriminating panel to detect staleness. Tests on bag packed coffee showed a difference in freshness between bag and vacuum packs at the first sampling (three weeks) when brews were prepared by steeping, but seven weeks were required when percolators were used in brewing.

Moisture absorption by bag packed coffee in the range above 7 percent is associated with serious loss of flavor. Below 7 percent moisture, variations in moisture level are not reflected in flavor changes specifically attributable to the moisture. Four weeks summer storage produced stale flavor, but eight weeks were required in the winter. Tests in this case were on steeped brews.

Another packaging development which is being watched with considerable interest by the coffee industry is the pressure packed cans which are now being distributed by one coffee manufacturer. This is not a new or revolutionary idea and it has been tried a number of times in the past by smaller companies without the advertising backing and support which is being given to the present marketing picture. Because of the very fine job done by the canning industry in convincing the housewife that a swelled can is a bad can the coffee industry is constantly bothered with complaints due to swelled coffee cans. Actually, the swelled coffee cans might be said to be an indication of highest quality, since it indicates that that particular package is definitely tight and having once been evacuated is simply being swelled by the escape of carbon dioxide from the coffee packed therein. The present pressure pack can aims to take advantage of this situation.

Another type of package in which there is equal interest is the development now being carried on by one of the packaging manufacturers. It is aimed at packing coffee under vacuum in flexible containers. Should such a development be successful, it would represent a real, competitive challenge to the present can pack since it would be anticipated that appreciable saving could result therefrom.

One thing of a practical nature that might be added here is that those who use coffee at home will find it advantageous from the quality point of view to store open vacuum tins of coffee in the refrigerator for retention of optimum quality. Coffee purchased in paper bags will better retain its original quality if placed in tightly closed glass containers and placed under refrigeration.

One of the processed coffee items which has been neglected thus far is decaffeinated coffee. You might be interested to know that this is an excellent example of chemical engineering applied to the food industry. In addition to providing those people who are sensitive to caffein with a palatable coffee beverage, it also provides the pharmaceutical and beverage industries with appreciable quantities of caffein.

At the time General Foods purchased the rights to the Roselius decaffeinated coffee process, this particular operation included a rather complex chemical engineering type treatment of coffee requiring longer than 86 hours of actual processing. Without attempting to go into too many details, a comprehensive study of the operation over the last ten years has brought about marked advances. The cycle was first reduced to 68 hours; then to 48 hours, then to 36, and eventually to 24 hours. At that point the batch operation gave way to research developments and the process was placed on a continuous basis, using a liquid-liquid type extraction operation, which has since been completely patented. For this purpose it was found by laboratory experiments that water was a much better solvent for caffein and could remove the caffein from the coffee in 6 to 8 hours. The product that resulted was distinctly better than any product made before. This, to our knowledge is the first application of liquid-liquid extraction techniques in the food industry and resulted in the development of some rather novel equipment. The final result is that the product has been changed from a beverage having a decidedly inferior taste when compared to fresh coffee to one that is almost indistinguishable in quality from fresh coffee.

Mechanically, instead of a 24-hour batch extraction cycle we have a continuous 8-hour extraction cycle. Solvent losses have been reduced to a minimum and many operating economies have been established.

Caffein yields and recovery of caffein have been markedly increased which represented a real contribution during the last war. In fact solvent losses were reduced by 75 percent and caffein recovery was just about doubled.

COFFEE EXTRACTS

This leads us to the "baby" of the coffee industry, namely, the entire field of coffee extracts. The phrase coffee extracts is used instead of powdered or "soluble" coffee because of the recent introduction into this field of the frozen liquid coffee concentrates. Frozen liquid coffee is an entirely new development, not more than a few months old inasfar as its market appearance is concerned. There is obviously a considerably longer span of time behind the research and introduction of the product. Its introduction has been stimulated by the marked success of orange juice concentrates.

The process is relatively simple in that concentrates are made by any number of industrial brewing or percolation processes. The concentrate is then packed in small tin containers and distributed in frozen form to the consumer. The product has the advantage of ease and simplicity of use and of low storage temperatures for maximum retention of character until it gets to the consumer. It is too new as yet to predict the size of the baby. It is possible, however, that it will provide a springboard for getting a better quality than has yet been envisioned in the coffee powders, if this better quality is not offset by higher costs and inconvenience of use after the can is opened.

In regard to powdered coffees or soluble coffee products, the dream of a soluble coffee has been with the industry for many years. However, we perhaps set our sights too high in that most of the coffee processors refrained from introducing such a product because they felt that the items they had developed did not completely duplicate freshly brewed coffee. It was left to one organization to pioneer this field and to do a remarkable job convincing the coffee manufacturers that the customer would accept certain shades of differential of quality in an effort to get convenience. Today, it appears that every soluble coffee manufacturer is striving to duplicate freshly-brewed coffee while at the same time selling an eminently satisfactory product which is being accepted by the consumer as a pleasant and satisfactory beverage, and in isolated instances, according to recognized consumer test procedures, actually more acceptable to the consumer than brewed coffee. A fact in this regard that is interesting to note is that a comparison of soluble and freshly brewed coffees conducted recently by the Armed Services showed no significant preference between the brewed coffee offered at mess and a number of commercial soluble coffees, with which it was compared (or paired).

While this development has taken place within the last 20 years, the process in oversimplified form can be said to involve the brewing of roasted, ground coffee, the concentration of such a brew to a point where it can be economically fed to a dryer followed by the drying of the concentrate by either spray or drum drying procedure. However, that is *really* an oversimplification. Having previously indicated that coffee is very susceptible to oxygen exposure, this criteria must be taken into consideration during the processing. Since coffee is a very delicate flavor in any event, improper handling of the brew at any stage of the operation results in loss of quality. Today most soluble coffee is being manufactured by some form of counter-current extraction system and the handling from the time the extract is separated from the grounds to the time the product is ready for packing in finished dry form is expedited in every way possible. Much work has been done in this direction both with regard to the design of extractors and the design of the dryers. Two companies have applied variations of the low-temperature blood plasma drying techniques to the operation and one of the products now on the market is made by a process involving continuous high-vacuum belt drying where the vacuum is maintained at a total pressure of less than 4 mm. of mercury by means of a steam pump and a water vapor absorption system. The surface of the belt is heated to 150-200° F. (66-93° C.) which causes immediate foaming and solidification without freezing. After six minutes exposure the film is reduced to less than 4 percent moisture; it is then scraped off ready for packing. This coffee unlike other products is instantly soluble in cold water as well as hot.

Another company is said to be working on a dryer to accomplish the same thing with improved economies of operation.

One process used on a small scale during the war and now being given some study by industry involves the concentration of weak brews by freezing techniques which have been developed in Germany prior to the war.

In some instances, corn syrup and other kinds of non-coffee carbohydrates have been added to the material, thereby reducing the degree of concentration and exposure necessary, in order to get a product having more flavor quality and less susceptible to the influences of moisture and other types of deterioration changes. Actually, this addition is still the subject of considerable argument.

In recent months attention is being paid to sanitation as demonstrated by the bacteriological count of the coffee extract in soluble coffee production. The standard plate count gives the number of bacteria per gram. For instance, in one company, the tentative acceptable count has been set at 100 per gram. This figure is being bettered and very often only 10 per gram are found.

A new market for soluble coffee has been established recently in the vending machine field. The machines are using powder, liquid concentrate, frozen concentrate and pellets. The problem of dispensing a cup of freshly brewed coffee is being worked on, and the possibility of this type of machine being the one of the future is pretty good.

Increased coffee costs are not a major problem in this field because the cost of the double-wrapped cup is the largest item in the cost of vending a cup of coffee.

What to do with waste grounds presents a minor problem in the coffee industry. Generally they are thrown away and in cases where they are hauled away, it amounts to an additional expense. One possible use is being developed whereby coffee grounds containing 65 percent moisture are burned as fuel.

Another possibility (patented) is the solvent extraction of the residual caffein and coffee oils in the grounds. This extracted product after autoclaving with aniline becomes an acceptable plastic filler.

FUNDAMENTAL RESEARCH

The commercial developments already cited have been aggressively paralleled by fundamental research. General Foods alone has a continuous record of over 25 years of unbroken effort in this field. Emphasis has shifted from time to time but always one objective has been kept in mind, to learn more about the changes that take place during the processing of coffee and about the physics and chemistry involved in those changes. Coffee flavor or coffee aroma constitutes one of the most expensive chemicals in the world. One may calculate the price of organic material in coffee aroma at about $2500/lb. Since this may contain 10, or 1, or 0.1 percent of essential constituents, the price per pound for effective flavor material may be $25,000, $250,000 or $2,500,000.

Some of the main volatile constituents of coffee are as follows:

Organic Compound	Approx. mg./gm.
Pyridine	200
Furfural	85
Aldehydes (mainly acetaldehyde)	30–125
Diacetyl Carbinol	15
Acetylmethyl Carbinol	15
Acetone	30–85
Phenols	30–110
Hydrogen Sulphide	3.0

We can account for about 95 percent of green coffee extract in terms of fairly well defined chemical classifications, proteins, ash, acids, etc., but when we come to the extract of the roasted product, "soluble coffee," we cannot account for more than 30 percent in terms of known chemicals and of this 30 percent, about 20 percent or two-thirds, is ash.

Today the roasting of green coffee extract is being carried on in our laboratories as a tool for detecting these individual constituents and observing the changes that take place.

Thermal studies are telling us more of the changes in heat balance occurring during the roasting process.

An effort to identify the constituents in coffee or aroma which give coffee character is progressing. This work has resulted in the theory that the sulfur compounds in coffee were the more important contributors to flavor. This idea has not changed in twenty years except for the modification that it is probably the lower boiling sulfur compounds that are important; the higher boiling ones being free of flavor or representing reaction products of the lower boiling compounds.

From studies on the roasting of green coffee beans, the theory was developed that pressure was formed in the beans during roasting and that pressure was necessary for the proper development of coffee flavor. This idea is still held today, the only qualification being that it is probably not pressure per se that is the essential force, but that the pressure indicates the necessity for keeping initial breakdown products together until the proper stage of roasting is reached when they can react with each other to make coffee flavor. A fourth idea is that the precursors of flavor and aroma are water soluble or water dispersible in the light of the elimination of flavor upon clarification or filtration of green coffee solutions.

Considerable effort has been directed to an explanation of the pH changes in coffee during roasting. It has been observed that green coffee starting at a pH of 5.8 drops during the roast to about 4.8 and turns around and rises again; the optimum roast being shortly after the turn. Several possibilities suggest themselves to this change including:

1. The formation and volatilization of acetic acid from the decomposition of the sugar.
2. The decarboxylation of acids formed by rearrangement of the sugar molecule.
3. The decarboxylation of chlorogenic acid.

It has been found that chlorogenic acid contributes a certain amount of body and astringency to coffee beverage; its decomposition gives some of the minor aromatic components of the aroma; and most important, it appears to be the best buffer acid thus far located in the coffee system. Sucrose appears to contribute the greatest amount of chemical reaction during roasting since it is completely decomposed. Sucrose reactive products contribute much of the color of the beverage, plus some aroma, bitterness, and sourness. Coffee proteins present a rather peculiar picture; purified protein appears to be perfectly stable to roasting. On the other hand the crude protein fractions seem to be the major source of coffee aroma. The inference is that the aroma is derived from minor protein components of a highly reactive nature. The trigonelline is known to generate pyridine during roast, and it may thus be responsible for some of the objectionable flavors in coffee which one has to, as we say, "develop a taste for." Caffein in coffee has no other function than to contribute to the bitterness.

That is about the substance of our knowledge of the fundamental components of coffee and the part they play in its being our best liked beverage.

In summary we have seen that the green coffee situation in Brazil is undergoing some rather drastic changes, both in areas and methods of cultivation in order to increase the productivity of each individual tree. It can be anticipated that the pace of this development will increase as time goes on.

From a processing point of view, the roasted coffee industry is now seeing the development of large scale continuous equipment and controls to provide a uniform product, which have not been available heretofore.

In the field of new products, the frozen liquid coffee is still in its infancy and powdered or soluble coffee has come to take an important place in the market. These two items raise new problems in equipment design, bacteriological controls, and waste disposal problems, which the industry has not had to deal with heretofore.

In the field of fundamental research, we are constantly striving to increase our knowledge of what happens during the roasting of coffee to produce the flavor which we all find so satisfying at the breakfast table. Thus far, progress on this most difficult problem has been rather limited. However, the coffee bean is slowly yielding its secrets to the impact of scientific work now going on.

A High Temperature Process for the Extraction of Concord Grape Juice[a]

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A method has been described for the preparation of juice from Concord grapes in a steam retort. Processes of five to one hundred minutes were used at temperatures from 190-250° F. (88-121° C.). The juice was removed from the heated atmosphere as it was formed and that remaining in the cooked grape mass was extracted in a basket centrifuge. Chemical analyses of juices produced by this process indicated that the times and temperatures used for processing may be varied over the ranges investigated without serious loss of juice quality. Triangular difference palatability tests indicated that grape juice processed at high temperatures in the absence of air is comparable in flavor to commercial juice.

In the commercial preparation of juice from Concord grapes, the fruit is harvested at full maturity to obtain the maximum of color and flavor. Processing is ordinarily carried through without delay to avoid the quality losses which result from holding the raw product for extended periods. Although Hartman and Tolman *(3)* recommended a holding period of 24 hours prior to processing, this procedure is not generally used.

In the conventional preparation of juice from Concord grapes, the stemmed and washed grapes are pulped, heated to approximately 160-165° F. (71-74° C.) and pressed in a rack and cloth press. The resulting juice is flash heated to 185-200° F. (85-93° C.), flash cooled to 30-35° F. (—1.11 to +1.67 ° C.) and stored at 26-28° F. (—3.33 to —2.22° C.) for 4-6 months to allow crystallization of the argols and sedimentation to take place *(1)*. The juice is then separated from the sediment, bottled, re-pasteurized and marketed.

The temperatures used prior to the pressing operation and subsequently for pasteurization are usually well below the boiling point of water *(1, 3, 5)*. This practice safeguards the color, aroma and flavor of the juice. In the pressing operation, however, there is considerable contact with the oxygen of the air which has been found to be detrimental to juice quality *(5)*.

In an effort to minimize contact with air during processing and eliminate the use of a press for extraction of juice, a process has been developed in which the juice is extracted by means of heat in an atmosphere of steam.

EXPERIMENTAL PROCEDURE

The annular cylindrical vessel used was 22 inches high and 22 inches in overall diameter with a four-inch space between the two vertical walls (Figure 1). The bottom was cut to circular shape from a sheet of 28-gage stainless steel and a circular center of 12-inch diameter was trimmed out. The edges were brazed with silver solder to the inside and outside sheets to make up the sides of the vessel. The edges of the vessel were reinforced at the outside surfaces with angle iron supports to facilitate handling with a hoist.

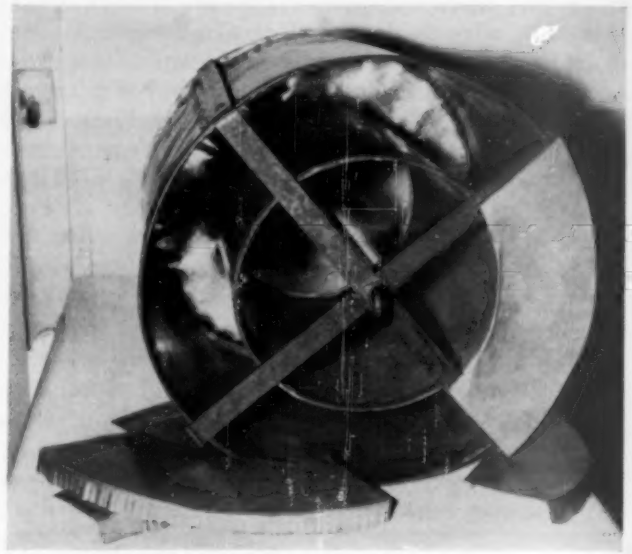

FIG. 1. Annular processing vessel.

The distance from the center of the vessel to either side was two inches. The annular space between the vertical sides was adequate to accommodate 50 lbs. of unstemmed grapes. A cover in four segments was fitted loosely to this doughnut shaped annular vessel. Copper-constantan thermocouples were mounted along the vertical center line of each batch of grapes at distances of one, two, three and four inches from the bottom of the vessel to record temperature changes during processing.

Grapes were harvested from the Hortitculture Farm at Ames in the morning and were processed within 24 hours of harvest.

Thirty-pound batches of grape clusters were heated in the annular vessel for various intervals of time at retort temperatures of 190°, 205°, 220°, 235° and 250° F. (88°, 96°, 104°, 113° and 121° C.). Each treatment was repeated once. To minimize the time of exposure of the juice to the elevated temperatures, the juice draining to the bottom of the annular vessel was periodically forced into a receiving vessel located outside of the retort. The steam pressure within the retort was used to force the juice out through a one-half inch stainless steel tube. Carbon dioxide gas furnished the pressure to force juice to the outside when the steam pressure in use was insufficient to accomplish it. The term "heat extracted juice' has been used to distinguish between juice forced from the processing vessel during processing and "centrifuged" juice which was recovered in the basket centrifuge after processing was completed. The rate of juice removal was controlled by a one half inch, silver-plated plug valve.

The hot juice was bottled in one quart juice bottles, sealed with crown caps, and pasteurized by heating at 185° F. (85° C.) for 30 minutes.

Three lots of the bottled samples were stored on open shelves at room temperature, and at the controlled temperatures of 70° F. (21.1° C.) and 40° F. (4.4° C.), respectively.

Analytical Methods

Analyses were performed on each batch of juice processed in an effort to establish the nature and extent of the chemical differences resulting from various heat treatments. Total

[a] Work on this project was made possible through a grant from the Council Bluffs Grape Growers Association, Council Bluffs, Iowa.

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acidity, tartaric acid, pH, density, viscosity, and tannin and coloring matter were determined.

Total tartrates were estimated colorimetrically by the metavandate method *(7)*. The pH measurements were made using a Leeds and Northrup glass electrode instrument and titratable acidity was determined by titration with sodium hydroxide to pH 8.25. Acidity was calculated as tartaric acid.

Soluble solids were determined by using the Abbe refractometer and relative viscosity was measured using the Ostwald viscosimeter.

The method of Hartman *(2)* was used for the estimation of tannin and coloring matter with an additional precaution found essential since the temperature of the solution appeared to affect the potassium permanganate titration. For the 100 ml. grape juice samples and the temperature range used here, this increased the titration by 0.15 ml. of 0.057 *N* permanganate per degree Centigrade. To standardize the procedure, a temperature of 33° C. ± 1° C. (92° F.) was chosen. Tartaric acid and other substances present in the grape juice were observed to show increased reactivity with the permanganate at elevated temperatures; hence, the higher the solution temperature, the greater the estimated tannin value. No error due to temperature changes was noted when pure tannic acid was titrated.

To be able to determine whether any of the methods for preparation of grape juice used in the laboratory produced detectable differences in the product and to guide future lines of inquiry, a taste panel of five judges was selected on the basis of ability to discriminate flavor in grape juice. To be a part of the taste panel, each judge was required to identify differences in a triangle taste test *(6)* made up of two samples from one treatment and one sample from a different treatment and to be correct in his selection in a minimum of five out of seven trials. For additional experience the judges were subjected to several practice trials to observe differences in grape flavor.

RESULTS

Experience indicated that use of the annular vessel allowed thorough and uniform heating of the grape mass in all but the shortest processes. It was observed that considerable quantities of juice were held mechanically in the cooked grape mass. This juice was removed by use of a small basket centrifuge. In calculating yields, corrections were made for the presence of stems in the raw product and the added weight derived from condensed steam. Yields ranged from 69.6 to 87.9 percent for all processes with an average of 82.3 percent. The inclusion of the average percentage of condensed steam accumulated during processing (5.1 percent) would raise the average yield to 87.4 percent.

Average weekly yields (Table 1) indicate that yields increased with advancing maturity from 74 percent

TABLE 1

Influence of Harvest Date on Yield of Grape Juice Made by Three Processes

Harvest week	Process temperature (F.) 220°	235°	250°	Weekly Ave.
	Process time (min.) 15	10	5	
	%	%	%	%
1	78.8	74.3	69.6	74.2
2	81.9	78.3	83.0	81.1
3	84.9	86.1	86.2	85.7

during the first week to almost 86 percent during the third week of the season.

In an effort to describe the relationships among times and temperatures of processing and the resulting yield, the areas under the heating curves were measured with a polar planimeter. The areas were measured from a temperature of 150° F. (66° C.) in each case. Effects due to differences in maturity were minimized by comparing processes which were completed within a period of three days (Table 2). No trends in yield were noted which might be attributed either to time or temperature of processing except a very slight increase in yields with increased processing temperature.

The average yields for all processes (Table 3) show increased juice yields directly related to processing time

TABLE 2

Relationship Between Extent of Heating and Yields of Grape Juice

Date	Time, min.	Temp., ° F.	Extent of heating [c]	Yield,[d] %	Ave. Yield, %
Sept. 9	15	220	2.20	81.8	
Sept. 9	30	220	11.37	83.8	81.2
Sept. 9	45	220	13.26	78.0	
Sept. 8	10	235	1.90	78.3	
Sept. 8	20	235	6.89	83.2	82.1
Sept. 8	30	235	12.31	84.8	
Sept. 9	5	250	3.41	83.0	
Sept. 7	10	250	6.42	84.2	83.6
Sept. 7	15	250	11.51	83.7	

[c] Expressed in terms of the area in square inches, measured from 150° F. (65.6° C.).

[d] Corrected for presence of stems and for condensate.

TABLE 3

Effect of Varying Temperatures and Times of Processing on Yields of Grape Juice

Time, min.	Retort temp., ° F.	Average yield, %
80	190	76.12
100	190	77.87
40	205	76.79
60	205	80.80
80	205	83.72
15	220	82.62
30	220	84.93
45	220	84.21
10	235	79.57
20	235	84.20
30	235	84.94
5	250	79.57
10	250	85.55
15	250	85.27

[e] Corrected for presence of stems and for condensate.

and processing temperature. Under the conditions imposed in the use of the high temperature extraction process, juice yields of approximately 85 percent were obtained in 30 minutes at 220° F. (104° C.), in 20 minutes at 235° F. (113° C.) and in 10 minutes at 250° F. (121° C.). Additional processing time at each of these temperatures produced no marked changes in yields.

In some trials, the rate of accumulation of juice forced from the retort was determined by time-volume measurements (Table 4). While no clear-cut differences were

TABLE 4

Time Required to Accumulate Juice Forced from the Retort During Processing

Forced juice yield, ml.	Heating temperature 220° F. (3 trials) min.	235° F. (3 trials) min.	250° F. (2 trials) min.	Ave., min.
1350	6.08	8.00	5.75	6.61
2450	7.25	9.42	7.13	7.93
3625	9.00	12.50	10.38	10.63
4865	13.00	15.14	13.25	13.80
6205	10.92	19.08	16.63	15.54
7575	17.83	17.66	18.88	18.12

noted which might be ascribed to specific processing time-temperature combinations, the average values for eight trials at three temperatures indicate that the rate of formation of the forced juice decreased as the process progressed. The curve describing this relationship is plotted in Figure 2.

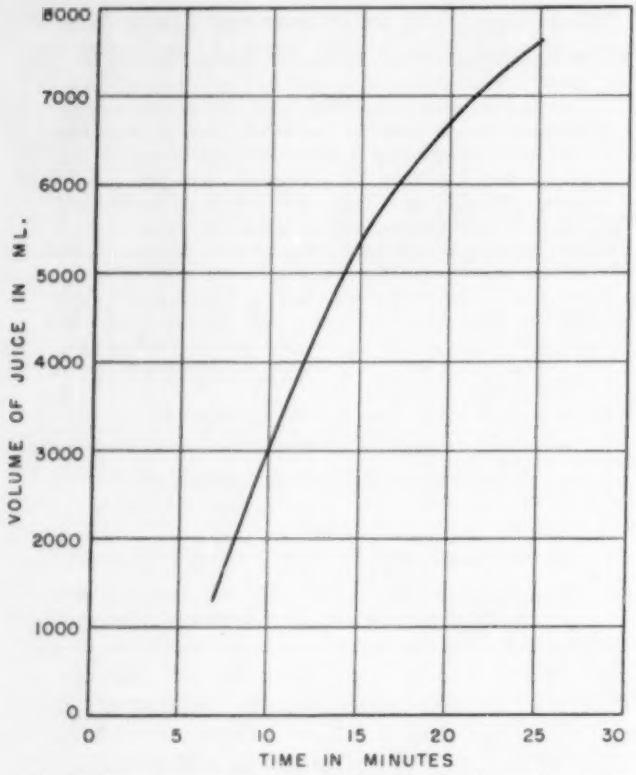

FIG. 2. Average rate of accumulation of juice forced from the retort during processing.

After the first week of harvest the values for total acidity expressed as tartaric acid remained close to 1.0 percent (Table 5). The weekly averages were 1.13, 1.01 and 0.94 percent. The total tartrate values showed no consistent trends with maturity, remaining near 1.2 percent throughout the harvest. The pH was observed to increase slightly with each succeeding week of maturity from 3.27, 3.37 to 3.41. The values for refractive index changed very little with maturity. Viscosity values were apparently affected both by the variations in pectin content and by the increase in sugar content.

TABLE 5

Affect of Harvest Date (Maturity) on Chemical Composition of Heat-Extracted Juice [1]

	Week of the season		
	First	Second	Third
Tartaric acid, percent	1.13	1.01	.94
Total tartrates, percent	1.18	1.26	1.17
pH	3.27	3.37	3.41
Refractive index	1.3545	1.3561	1.3555
Relative viscosity (Ostwald), seconds	607	479	557
Tannin and coloring matter, percent	0.25	0.28	0.31

[1] Weekly averages of all process times and temperatures.

In the late maturity periods, the sugar accumulation in the grapes increased the viscosity somewhat, compensating for the loss of pectin in the juice, and causing the viscosity values to be confounded by the two opposing effects.

Analyses demonstrated that maturity had a slight effect on the amount of tannin and coloring matter, increasing the value from approximately 0.25 percent during the first week, to approximately 0.31 percent during the third week. Only a slight effect due to variations in the treatment was noted.

An interesting observation at this point is that the tannic acid values were not abnormally high with any of the higher temperatures of extraction in spite of the inclusion of stems with grapes during processing. This is in contradiction to previous statements *(1, 4)* that low temperature processing methods using about 150° F. (66° C.) are preferable because elevated temperatures excessively increase the tannins in the juice of some varieties of grapes.

Chemical composition remained relatively constant over the range of processing temperatures used (Table 6). Results indicated that juices processed by the high temperature extraction method compared favorably with commercial juices.

TABLE 6

Affect of Processing Temperatures on the Chemical Composition of Heat-Extracted Juice [1]

	Processing temperatures				
	250° F.	235° F.	200° F.	205° F.	190° F.
Tartaric acid, percent	1.02	1.00	1.01	1.01	0.87
Total tartrates, percent	1.26	1.21	1.11	1.29	1.07
pH	3.31	3.37	3.39	3.39	3.50
Refractive index	1.3554	1.3563	1.3558	1.3547	1.3550
Relative viscosity (Ostwald), seconds	530	512	594	502	430
Tannin and coloring matter, percent	0.29	0.28	0.28	0.28	0.27

[1] Averages of all samples processed at a given temperature.

The grape juice having the most desirable color was observed to be that stored at 40° F. (4° C.) rather than that stored at room temperature. In all cases the color of juices stored at 90° F. (32° C.) deteriorated most rapidly, and in some cases the color of juices stored at the higher storage temperatures changed from dark red to orange red.

The juices recovered in the basket centrifuge were stored at 70-90° F. (21-32° C.) and were blended subsequent to storage with the heat extracted juice in proportion to original yields. These blends were observed to retain the same amounts of total tartrates as the separate fractions of the juice. Most important of all, the centrifuged liquor apparently can be blended with the heat-extracted juice into a product possessing a flavor similar to that of the heat-extracted juice.

The three important causes of the color variation observed visually in juices prepared by the high temperature process were maturity, processing temperature and storage temperature.

Increases in depth of color were apparent in each case with advancing maturity. Fairly clear juices predominantly purple in color resulted when mature grapes were processed at low temperatures. However, the low processing temperatures tended to decrease yields.

The influence of processing temperature on color was obvious upon a visual observation of samples prepared

from grapes which were harvested on the same date. Process temperatures employed were 250° F. (121° C.), 235° F. (113° C.), and 220° F. (104° C.). It was noted that the juices resulting from high temperature processes were fairly red in color, while those from low temperature processes were purple.

Results of storage tests conducted at room temperature indicated that, as the storage time increased, the color of the juice changed to a blue-purple and part of the suspended solids and tartrates settled out.

Juice stored at a low temperature (40° F., 4° C.) was observed to retain its red color for a period of at least eight months, the duration of the test.

By visual comparison it was observed that juice stored at 40° F. (4° C.) showed a marked retention of bright (red) color which was not apparent in similar samples stored at 70° F. (21° C.). No difference in color was noted between "heated extracted juice" and juice subsequently extracted from the cooked grapes through use of the basket centrifuge.

Suspended material was first noted in juice emerging from the processing vessel. This juice was crystal clear while it was hot but became cloudy as the temperature decreased, probably due to the flocculation of pectinous materials. Upon storage, sedimentation continued and may have been a major factor in the color changes noted.

SUMMARY

A process has been outlined for the extraction of juice from Concord grapes by means of heat in the absence of air.

Processes at 190-250° F. (88-121° C.) for five to 100 minutes generally allowed the recovery of approximately 85 percent of the raw grape mass as juice when the mechanically held juice remaining in the cooked mass was removed with a basket centrifuge.

Determinations of total acidity, tartrates, pH, refractive index, viscosity, and tannin and coloring matter in juices from grapes subjected to the various time-temperature combinations used in processing revealed the non-critical nature of the process.

The inclusion of stems or to the higher processing temperatures employed did not result in higher tannin contents.

Triangular difference palatability tests indicated that grape juice processed at high temperatures in the absence of air is comparable in flavor to commercial juice.

Increased maturity, decrease in processing temperature and low temperature storage all aided in obtaining a desirable color in the juice.

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Biological Methods of Blood Removal and Their Effectiveness in Reducing Discoloration in Canned Dungeness Crabmeat[a]

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The bleeding of crabs (Crustacea) for the purpose of improving the quality of the canned product was studied. Bleeding was found to be beneficial. Experimental data and a discussion of results are given.

INTRODUCTION

This study is concerned with the Dungeness crab *(Cancer magister)* which is harvested along the coastal waters of Oregon, Washington, Northern California, British Columbia, and Alaska. This crab is used to a large extent in canning. In this procedure many unfavorable changes take place in the meat. These changes are: (A) the formation of a blue-gray color usually found at the ends of the muscle strands of the body meat and on the leg sections, (B) a general browning throughout the can content, (C) apparent flavor impairment and (D) a slight texture change.

A search through the literature reveals that this blueing is associated with the copper in the hemocyanin *(12)*, the oxygen carrying constituent of crustacean blood *(11)*. Takajasu and Fukuhara *(12)* proposed that this blueing is a sulfide derivative of hemocyanin and suggested the formula R-Cu-S-Cu-R for it, where R stands for a protein molecule. Motohashi *(12)* claims to have reproduced this blue coloration by heating crab blood in air at various temperatures for 10 minutes. According to his experiment, cooking between 176-194° F. (80-90° C.) produced

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blue spots within one hour. Fellers *(2)* proposed that this blue is due to the formation of copper-ammonia complexes and to sulfides to some extent.

Even though various chemical, physical, and bacteriological methods for the elimination of blueing have been reported in the literature *(1, 2, 3, 4, 9)*, further investigation on this problem was thought necessary, as none of these methods are now employed with complete satisfaction by the industry on the Pacific coast.

While various methods or practices having to do with the partial elimination of the blood have been used in crab canning throughout the world, the purpose of this experiment was to derive a satisfactory biological method of a more complete blood removal to increase the organoleptic values of the finished canned product.

EXPERIMENTAL PROCEDURE AND RESULTS

Bleeding Methods

The Japanese claim to have lessened blueing in canned King crab through methods eliminating some blood from live animals *(12)*. At the present time some Pacific Coast canners use a butchering method for crabs which will remove some blood. This method consists of pulling the carapace off the crab, breaking the body in two, shaking off the viscera, and removing the gills with the thumb. Immediately after steaming or precooking in a water bath, large quantities of blood coagulum are evident which cannot be removed completely by any washing procedure now in use. This method was considered insufficient for maximum removal of blood by this Laboratory.

Considering the crustacean and more particularly this crab's anatomy, two methods of blood removal from live crabs, employing the pumping action of the animal's heart, were examined.

Sternal Artery Method. The sternal artery is the large artery which descends from the heart and leads to the ventral thoracic and ventral abdominal arteries, running respectively anteriorly and posteriorly *(10, 11)*, supplying the various ventral appendages.

It was found that the sternal artery could be severed in live crabs. The crab was turned on its back and an opening, approximately ¼ inch in diameter and 1 inch deep was drilled beneath the tail in the area just below or underneath the junction line of the last two tail segments, and toward the heart. The crab was turned immediately onto the ventral side. A definite pulsating blood flow from this opening was correlated to heart action. Expiration time was 3-5 minutes. Quantities of blood removed varied from 20 to 75 cc., depending upon the accuracy of the drilling and the size of the crab. Upon precooking, blood coagulum was still evident, especially on the leg segments. However, the quantity was diminished from that found after use of the current commercial butchering method.

Removal of Appendage — End Method. Bleeding by this method depends both on heart action and drainage. When the ends of the immobile parts of the claws and the last segments of each walking leg were removed (in the area indicated by dotted lines in Figure 1) and the crabs were elevated so that the appendages hung down, a good pulsating bleeding action was observed. Blood coagulation or stoppage was not apparent on the injured legs. Self amputation (autotomy), which is a natural function preventing undue loss of blood from injured appendages among animals of this group *(11)*, did not take place. Blood removal was even better than in the sternal artery method, varying from 55 to 185 cc., depending upon the size of the crab. Expiration rate was from 2-3 minutes. The amount of blood coagulum found upon precooking was very insignificant, both in body and leg meats.

EFFECT OF BLOOD REMOVAL

Visual and Chemical Effects

A test was devised to determine the effect of blood removal, by the new bleeding methods described, upon the finished product. Using the copper concentration of crab blood as an indicator, determinations of the amount of blood remaining in cans prepared from bled and unbled crabs, and of whole crab blood were made. A visual classification of the amount of blue was given to the cans before the copper determinations were made to see whether or not there existed any correlation between the blue discoloration and the blood content, as had been claimed by the Japanese investigators *(12)*.

FIG. 1. Bleeding by appendage-end method. Claws and walking legs are removed within areas indicated by dotted lines.

For the copper determination a method employing sodium-diethyldithio-carbamate and a Beckman spectrophotometer was used *(7, 6)*. Samples were visually scored from A to D, with A being least blue and D most blue. On a dry basis the copper contents were obtained in the visually scored samples and whole blood. The results are shown in Table 1. These data show that there is a correlation between the blue-gray discoloration and the blood content of the canned meat, and that the blood content can be diminished less than half by the bleeding methods described.

TABLE 1

Visual and Chemical Determinations of the Effect of Blood Removal

Sample	No. crabs or cans	Visual score	Aver. Cu content
			%
Blood	15	.....	0.1050
Meat, unbled	12	C-	0.0109
Meat, bled	4	A	0.0042

ORGANOLEPTIC EFFECT

Further proof of the advantages of the bleeding was sought, and an organoleptic method was decided upon. In this test, unbled and bled crabmeat was canned in exactly the same manner and a taste panel was conducted to establish the variation between the cans.

Method of Canning. One hundred sixty pounds of live crab, landed at Warrenton, Oregon, on August 1, 1950 and taken from pots situated near the shore of Seaside, Oregon, was divided into two batches. The first batch was bled by the leg method already described and then butchered. The second batch, serving as unbled controls, was butchered by the usual commercial method. Both batches were precooked for 14 minutes in fresh water. After their removal from the water tank, they were water sprayed to cool and stored in a cold room overnight. On the following morning, the meat was picked, washed in fresh water, and placed into C-enameled ½ lb. cans. These cans were then retorted according to a commercial method.

The cans containing bled and unbled meat were then divided into two parts. One part of each lot was incubated at 99° F. (37° C.) for 1 month, the other part was stored at room temperature (approx. 75° F., 24° C.) for 1 month.

Taste Panel. A taste panel was then conducted by the Lorant and Wiegand *(5)* procedure. In this procedure, numerical values are given by the tester from 1 to 10 for each category, 1 being undesirable and 10 ideal. The scores of testers who show poor checks on duplicates in each quality category, such as texture, flavor, etc., are rejected for that specific category; the remaining scores are then averaged.

Five cans of each of the four variables, bled, unbled, bled incubated, and unbled incubated meat were tested and the scores for the 5 cans were averaged. The results obtained are shown in Table 2. The results of the taste panel were statistically

TABLE 2

Average Taste Panel Scores for Canned Crabmeat Samples

	Blue	Brown	Flavor	Texture
Bled	7.06	6.72	6.20	6.58
Unbled	5.00	4.94	5.14	6.25
Bled, incubated	5.68	5.43	5.30	6.68
Unbled, incubated	4.22	4.57	4.54	6.00

analyzed by means of the "t" test *(8)*. On the basis of the grading method used, the decrease in blueing upon bleeding was found to be very significant (the observed difference could occur due to chance alone only 1 time in 100). The browning decrease was found to be significant (the observed difference could occur only 5 times in 100).

Drained Liquid Effect. The cans (5 of each) used in the organoleptic tests also showed a definite reduction of drained liquid. Meat packed by the commercial butchering method contained 17.2 percent free liquid upon a two minute drain. Leg bled and then butchered meat contained 13.2 percent free liquid based upon gross can contents. This fact can be explained when the moisture contents of crab blood and meat are taken into consideration. The total moisture content of the blood was found to be 94.54 percent *(7)*, while the total moisture content of the meat has been reported as 76.6 percent *(13)*.

DISCUSSION AND CONCLUSIONS

In the canning of Dungeness crabmeat, difficulties with discoloration in varying degrees have continued to be encountered by industry for one reason or another. From past experience with industrial practices and after study of some of the literature pertaining to various crustacean forms, the possibility of bleeding crabs to eliminate some of these troubles was considered. The results of this study showed that it is definitely advantageous to use a good method of blood removal from live crabs, in addition to the butchering method now employed and previously described.

It is common knowledge that the different kinds of animals processed by the meat industries are bled for the purpose of preventing bacterial and enzymic action, and loss of bloom in the meat. The meat industry bleeds through the jugular vein, a large easily accessible vessel, from which the animal can properly pump its blood. Since crab blood constituents long have been considered largely responsible for the discoloration difficulties, a satisfactory method of bleeding crabs was sought. After a consideration of the crab anatomy, the large descending or sternal artery of *Cancer magister* was used for this purpose. It was tried in numerous instances but was found not to be as efficient in removing a greater quantity of blood as another method, appendage-end bleeding. As in the meat industry, the basic requirement for good bleeding of crabs is that the animal be alive so that the pumping action of the heart will empty the blood system as well as possible. For this purpose the canner has to receive the crabs in live condition from the fisherman, and keep them in such condition until bled. Since crabs can live in air for several hours as long as they are kept moist, it is usually possible for the fisherman to deliver his catch in fairly good condition. As long as crabs have water over their gills, they can breathe enough to stay alive until delivered to the cannery. Keeping them alive can be accomplished by throwing buckets of sea water over the catch periodically. Because the animals are very sensitive to heat, the catch should be kept cool in the summer. For this purpose a wet tarpaulin spread over the catch would probably serve. Live boxes, as used in the lobster industry, would be ideal. In some preliminary tests in the laboratory, crabs were kept alive in aerated tanks, and when canned without acid, produced excellent packs. Since methods of keeping the catch alive depend upon the prevailing conditions, these should be worked out between the canner and the fisherman.

Immediately after the crabs are received by the canner, they should be bled. It is recommended that the "Leg End Method" of bleeding crabs should be employed industrially. Such an operation could be performed by any satisfactory means of producing an opening at the appendage ends large enough to permit a good pulsating flow of blood. Cutting by cleaver, scissors, knife, saw, or other suitable means could be adapted. The crabs could then be placed on a narrow, slow moving belt, where the blood would drain in the 2-3 minute period found experimentally. The removal of the appendage ends should not in any way interfere with present picking procedures.

Regular commercial procedures for butchering, precooking, and canning should be used from then on. However, since bacterial decomposition or spoilage takes place in crustaceans extremely rapidly *(9)*, it is suggested that no storage procedures should be employed before the meat has been retorted. Acid treatment is still recommended as another precautionary measure to allay discoloration.

When this practice of bleeding is used by the industrial packer of crab meat, several advantages will become apparent. (a) The "blueing" so commonly found in the finished product will be diminished appreciably. (b) The other discoloration, that of "browning," which generally destroys the natural bloom of the meat, will be diminished to a minimum or eliminated altogether. (c) The coagulum or curd-like material (coagulated blood) which becomes evident after precooking and which appears in varying amounts in the finished product even though the meat has been washed, will be reduced appreciably. (d) By eliminating a great portion of the blood of the animal early in the procedure, there will not be the degree of contamination by the blood in any of the later phases of the canning operation, as there has been in the past. (e) The required drained weight of the finished product will be achieved more easily since the blood, bearing a larger quantity of moisture than the meat, has been removed prior to the precook. (f) The meat from crab so handled will produce a rela-

tively "dry" pack, which is preferred by the consumer. (g) When stored, packs containing bled meat will show less discoloration than unbled packs. (h) As in any new installation, bleeding will have its costs, initial and continuing, but these costs will be partially or wholly defrayed by the advantages derived therefrom.

SUMMARY

1. The sternal artery and appendage-end methods of blood removal in live crabs were investigated and compared, with the latter being more efficient.

2. The effectiveness of blood removal in reducing discoloration in canned crab meat was examined. A definite correlation between discoloration and blood content was found. Bleeding reduces the blood content in the canned meat to less than half.

3. Organoleptic tests revealed that bleeding improves the canned product in reducing the blue-gray discoloration, diminishing the general browning, improving flavor, and slightly improving texture.

4. The drained liquid was reduced by approximately 20 percent in cans containing bled meat.

Acknowledgment

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The Influence of Pasteurization, Freezing, and Storage on the Functional Properties of Egg White[a,b]

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The value of egg whites that had been subjected to heat pasteurization, different freezing temperatures, and different storage periods, was measured by angel food cake volume and score.

Pasteurized egg whites produced undesirable cakes unless the standard beating procedure was modified. The modified procedure produced satisfactory, but not optimum quality cakes.

Cake volume resulting with frozen egg whites was nearly as good as that produced with fresh egg whites, and judging scores were as good or better.

Bakers and others who use egg white occasionally obtain poor quality cakes and other food products in which frozen egg white is used. The reason for the poor results is seldom known but they are often attributed to the quality of white used.

Funk *(3)*, Murphy and Sutton *(7)*, and the Egg Producers Council of Australia *(2)* have advocated the pasteurization of eggs in the shell in order to preserve quality. It is claimed that heating eggs to 140° F. (60° C.) for ten minutes with constant agitation does not impair the functional properties of the white.

Winter, Greco, and Stewart *(11)*, Gibbons, Fulton, and Reid *(4)*, and Winter, Stewart, McFarlane, and Solowey *(12)* have pasteurized liquid egg products before freezing or drying in order to reduce the viable bacteria count, destroy possible pathogenic bacteria, and improve the keeping quality of the products.

Wilkins and Winter *(10)* and Slosberg, Hanson, Stewart, and Lowe *(9)* observed that heating egg white increased its beating time and decreased the stability of the foam. The latter workers *(9)* also reported that heated egg white produced lower cake volume. Miller and Vail *(6)* obtained equally good angel food cakes made with fresh and thin frozen white. Thick frozen white produced less desirable cake.

We have studied some of the possible causes of poor functional properties of frozen egg white. The influence of heat (pasteurization), freezing and freezing temperatures, and storage temperature and time have been investigated. The functional properties have been assessed by measuring cake volume and by judging panel scores of angel food cakes made from egg white treated in various ways.

[a] This study was made possible by a grant from the Refrigeration Research Foundation to the Ohio State University Research Foundation.

[b] Presented before the Tenth Annual Meeting of the I. F. T., Chicago, Illinois, May 23, 1950.

EXPERIMENTAL

In this study two lots of frozen and pasteurized egg white were compared with fresh egg white for making angel food cakes. The Lot 1 egg white consisted of two 30-pound cans of frozen white obtained frmo a local egg-breaking plant. The cans were defrosted at room temperature and mixed in a large tub. Half of the mixture was placed in quart freezer tubs, frozen at —20° F. (—29° C.) and stored at 0° F. (—18° C.). The remainder was pasteurized in an experimental pasteurizer, according to the method of Winter, Greco, and Stewart *(11)*, at 134° F. (57° C.) for four minutes. It was then packaged, frozen, and held like the unpasteurized portion. Nine dozen Grade A eggs were purchased at a market and held in a household refrigerator to serve as the fresh egg-white controls. The mixed whites from one dozen of the eggs were used as the source of fresh egg-white control at each baking.

Two cases of Grade A spring eggs were used for Lot 2 white. They were divided at random into three groups. Group 1 was oil-treated, cartoned, and held in a household refrigerator to serve as the source of the fresh white control. The other two groups of eggs were broken out, the whites and yolks separated, the white filtered through a Buchner funnel, mixed in a can, and separated into two portions. One was packaged, frozen, and held as the unpasteurized frozen egg in the same manner as Lot 1 white. The other portion was pasteurized under the same conditions as the Lot 1 pasteurized white before freezing and storage.

The bacterial analysis of the unpasteurized and pasteurized frozen white samples was made according to the method of the Association of Official Agricultural Chemists *(1)*, with the exception of the coliforms, which were determined by the method of Leifson *(5)*.

The white samples were tested for their value in making angel food cakes according to the method of Miller and Vail *(6)*. The cake formula used contained:

	Grams
Egg white	425.0
Cream of tartar	8.2
Salt	1.0
Sugar	300.0
Flour	100.0
Vanilla	4.0

The whites were defrosted in a household refrigerator and brought to 70° F. (21° C.) by placing the tub in a warm water bath.

A Kitchen Aid Model 4 electric mixer with a whip attachment was used for the beating and mixing operations. The whites were beaten for 30 seconds at speed number 2. In order to produce a more uniform foam condition in all three samples, the speed was increased to number 3 with the second lot of pasteurized frozen white.

The cream of tartar and salt were added and the mixture was beaten for 90 seconds. The beating time was extended to 150 seconds for the pasteurized frozen egg samples of Lot 2 eggs in order to produce a foam similar to that of the unfrozen white control.

The specific gravity of the foam was determined at this stage of the mixing process by dividing the weight of a tared measuring cup level full of foam by the weight of the same cup full of water. The cup of foam was returned to the mixing bowl after the weight was obtained.

Two hundred grams of sifted sugar were added, a tablespoon at a time at five-second intervals and within a minute, with the mixer operating at number one speed. The vanilla was added and the meringue mixture beaten for two minutes except for the pasteurized frozen white with Lot 2 eggs which was beaten for one minute only.

The flour, sifted with the remainder of the sugar, was added a tablespoon at a time at intervals of 10 seconds and within 90 seconds, with the mixer operating at number one speed. The mixing was continued for 90 more seconds except in the case of the pasteurized frozen white of Lot 2 eggs which was beaten for only 30 seconds longer.

Seven hundred grams of batter were baked in tube pans which measured 4.5 inches in height, 5.5 inches in diameter at the bottom, and 9 inches at the top. The cakes were baked at 350° F. (177° C.) for 40 minutes. The pans were inverted and cooled over night.

The height of the cakes was measured by means of a ruler. An index to the volume of the cakes was obtained by cutting a 0.5-inch slice, placing it on white paper, tracing the outer edge of the slice on the paper, and using a polar planimeter to measure the area.

The cakes baked with Lot 2 eggs were scored subjectively by five judges. Characteristics considered were appearance, texture, moisture, tenderness, and flavor. One cake was baked with each kind of egg from each lot on nine different occasions. The data obtained were treated statistically. The averages of the results obtained are summarized in Table 1, and Figures 1 and 2.

The original bacterial count was higher in the samples of commercial egg white than in the white from Grade A eggs. Pasteurization destroyed 90 percent of the standard plate and all of the coliform bacteria in Lot 1 eggs and more than 90 percent in Lot 2 eggs.

The pH of the white was not influenced appreciably by pasteurization and freezing.

The specific gravity of the foam, containing cream of tartar and salt, was about the same for unfrozen and frozen white (0.16 or 0.17). It was much higher in the case of pasteurized frozen white. Increasing the beating speed and time for pasteurized frozen white reduced the specific gravity of the foam from 0.37 to 0.25.

The lower the specific gravity of the foam and cake batter produced by a given sample of egg white, the greater was the volume and score of the angel food cake.

Unfrozen and frozen egg white produced equally good cakes. Pasteurized frozen white produced heavy,

TABLE 1

The Value of Fresh, Frozen and Pasteurized, and Frozen Egg White for Making Angel Food Cakes

Observation	Lot 1			Lot 2		
	Fresh Control	Frozen	Past. and Frozen	Fresh Control	Frozen	Past. and Frozen
Standard plate count bacteria (per g.)		467,000	21,900		27,000	83
Coliform bacteria (per g.)		0	0		2,000	10
pH of white	8.9	9.0	9.0	9.1	9.1	9.0
Specific gravity of foam with salt and cream of tartar, added	.17	.17	.37	.16	.17	.25[e]
Specific gravity of cake batter	.33	.38	.64	.31	.36	.44[e]
Height of cake (inches)	4.00	3.75	1.50	4.00	3.75	3.50
Area of cake, cross section (sq. in.)	11.9	10.7	5.3[e]	13.3	12.4	10.7[e]
Cake score—total—(possible 100)	Good	Good	Very poor	89	86	74
Appearance	Good	Good	Poor	91	85	68
Texture	Good	Good	Very poor	91	80	64
Moisture	Good	Good	Very poor	85	85	74
Tenderness	Good	Good	Poor	91	91	84
Flavor	Good	Good	Very poor	88	89	79

[e] Statistically significant at one percent level.

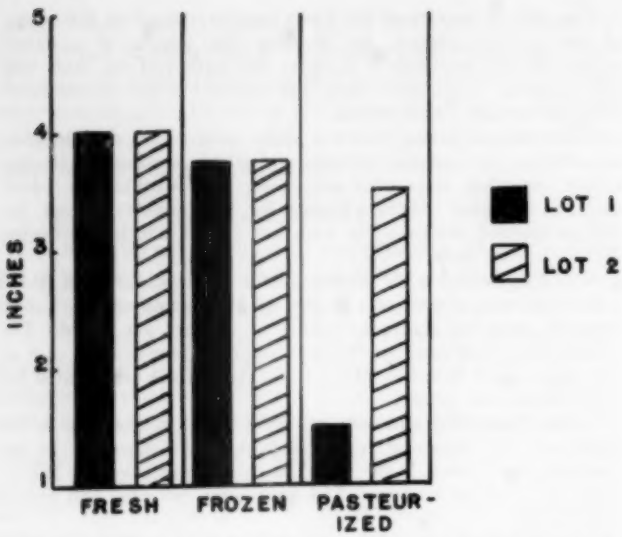

FIG. 1. Influence of fresh, frozen, and pasteurized and frozen egg white on height of angel food cake.

tough, coarse textured, wet, and objectionable cakes when the same beating time was used as for the unfrozen and unpasteurized frozen controls. Satisfactory angel food cakes could be produced, however, from these samples by increasing the beating speed and time through the meringue steps of preparation and shortening the time for mixing the batter.

AGE OF FROZEN WHITE

Some bakers prefer egg white which has been frozen and stored for six months or longer to that which has been frozen only a few days or weeks. They believe that the older white, commonly referred to as aged or seasoned, makes better quality angel food cakes than the new or recently frozen white which is frequently referred to as green white.

Seven cases of Grade A eggs were used for Trial 1 of the age study. They were produced in the spring by layers kept in confinement. The eggs were broken out and the whites and yolks separated. The whites were filtered through a Buchner funnel and mixed in a large container so as to avoid the incorporation of more than a trace of air bubbles. Ten-dozen pint freezer jars were filled three-fourths full with the white. Two dozen of the jars of white were frozen and stored at + 10° F. (—12° C.) and similar lots at 0°, —10° and —20° F. (—18°, —23° and —29° C.). One lot was frozen at —50° F. (—46° C.) and stored at —20° F. (—29° C.). A dozen Grade A eggs served as the source of the fresh white control each time that cakes were baked.

The samples were defrosted overnight in a household refrigerator and then brought to 70° F. (21° C.) in a water bath. The thick and thin whites were mixed by hashing in a Waring blendor for 30 seconds at greatly reduced speed to avoid the incorporation of air.

The cake formula and methods of mixing used by Slosberg et al. *(9)* was used in the tests. The angel food cake formula consisted of:

	Grams
Egg white	122.0
Cream of tartar	1.0
Salt	0.6
Sugar	125.0
Flour	45.0

The egg white was beaten five seconds in a Kitchen Aid Model 3-B electric mixer at high (No. 10) speed. The salt and cream of tartar were then added and beaten five seconds. Ninety-four grams of sugar were added in four equal portions at the end of 10, 15, 18.5, and 22.5 seconds of beating after which the mixture was beaten to a finish. This was the soft peak stage, i.e., where the meringue peak turned down slightly when the wire whip was removed.

The flour, sifted with the remaining sugar, was added to the meringue in four equal portions. After each addition, it was mixed in with 20 circular motions with a pastry blender, giving a total of 80 circular motions.

One hundred and twenty-five grams of cake batter were placed in each of two rectangular cake pans 5.5 x 3 x 2 inches, fitted with wax paper in the bottom. The cakes were baked at 350° F. (177° C.) for 32 minutes. The pans were inverted over wire grids and cooled over night. Cake volume was determined by the rape-seed displacement method. The surface above the cake was filled with rape-seed and leveled off with a spatula. The seeds were then poured off and the volume measured in a graduated cylinder. The volume of the cake pan minus the volume of the rape seed was considered as the volume of the cake.

The cakes were scored by a panel of five judges. Moisture, texture, lightness, and tenderness were considered by the judges.

Cakes were baked at the end of 1, 3, 5, 7, 9, 11, and 24 weeks storage. The data are summarized in Table 2.

A second trial was conducted with a different lot of eggs. These were from the same source as those in Trial 1, were of similar quality, and were prepared, frozen, defrosted and mixed as in Trial 1. The Slosberg et al. *(9)* procedure was modified slightly in this and succeeding tests for baking angel food cakes.

Sixty-one grams of the defrosted and thoroughly mixed, thick and thin white were weighed into a Kitchen Aid (3B) mixing bowl. The white was beaten at the fastest speed (No. 10) for three seconds. Three-tenths gram of salt and 0.9 gram of cream of tartar were added and the mixture was beaten at high speed

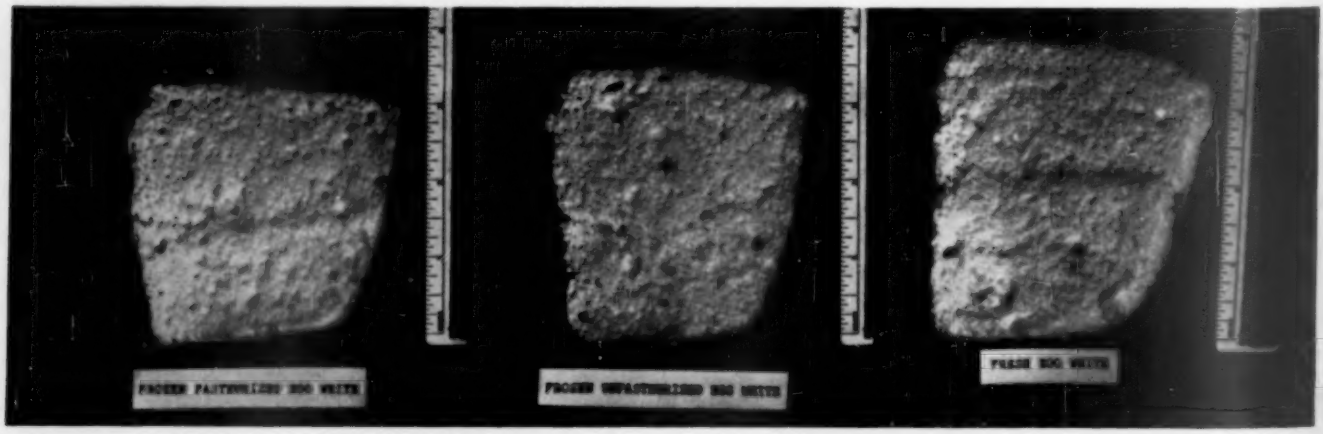

FIG. 2. Influence of pasteurization and freezing of egg white on the texture and volume of angel food cake.

TABLE 2

Influence of the Age of Frozen Egg White on Its Value for Making Angel Food Cake

Storage time, weeks	Trial 1 (Av. of 5 cakes)		Trial 2 (Av. of 8 cakes	
	Cake Volume (125 g. batter), ml.	Cake [d] Score	Cake Volume (85 g. batter), ml.	Cake [d] Score
0 (fresh control)	541	71	423	92
½	....	....	416	90
1	539	70	425	95
2	....	....	416	92
3	561	73	430	93
4	....	....	428	92
5	554	70	....	....
7	561	71	....	....
9	554	70	....	....
11	560	70	....	....
24	566	....	....	....

[d] Highest possible score = 100.

for three seconds. Forty-seven grams of sifted sugar were added in equal parts at the end of 6, 9, 12, and 15 seconds. The mixture was beaten to a finish (soft peak stage). A twice-sifted mixture of the flour and remaining sugar (15.5 grams) was added to the meringue gradually during 30 seconds with the beater operating at low (No. 1) speed. Eighty-five grams of batter were placed in an ungreased 500 ml. rectangular baking pan (2½ x 3 x 5½ inches) fitted with wax paper in the bottom. The cakes were baked at 350° F. (177° C.) for 27 minutes. The cake pans containing the cakes were inverted on a wire grid and allowed to cool. The volume was again determined by the rapeseed displacement method. The cakes were scored subjectively by a panel of five judges. The score was based on appearance, texture, tenderness, moisture, and flavor. Duplicate cakes were baked with samples frozen at four different temperatures. Cakes were also baked after the samples had been stored for 0.5, 1, 2, 3, and 4 weeks. The data are summarized in Table 2 and Figure 3.

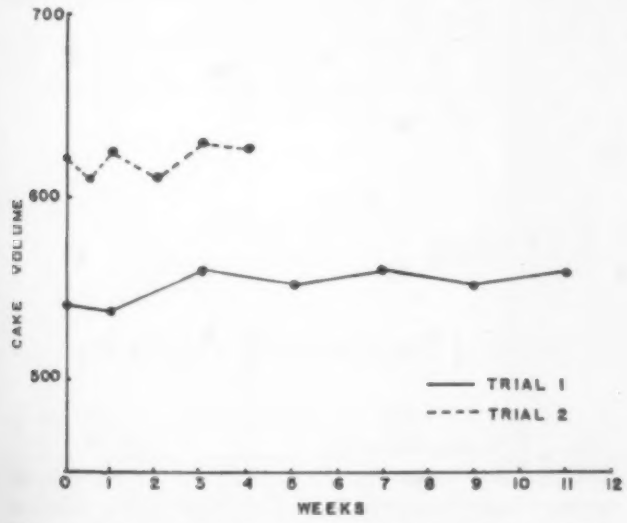

FIG. 3. Influence of age of frozen egg white on cake volume.

There was no appreciable difference in cake volume or score of cakes baked with fresh white, recently frozen white, or white which had been frozen as long as six months.

Our data are in agreement with that reported by Pearce and Lavers *(8)*. They found that mechanical treatment or freezing time did not affect the baking volume, foaming volume or pH of white, whether measured before freezing, after freezing, or after frozen storage.

FREEZING AND STORAGE TEMPERATURES

A few studies have been made on the functional properties of egg white before and after freezing. However, to our knowledge, studies have not been reported on the functional properties of egg white frozen and stored at various temperatures. The freezing temperature and the storage temperature and time have been suggested as possible causes of variation in the functional properties of different lots of frozen egg whites.

Part of the egg white samples prepared and frozen for the age studies with frozen whites (Trial 1) were used for a study of the influence of freezing temperatures and time. The method used by Slosberg, et al. *(9)* and previously described, was used for judging quality.

Duplicate cakes were baked with fresh white, and with white frozen at 10° F., 0° F., —10° F., —20° F., and —50° F. (—12° C., —18° C., —23° C., —29° C., and —46° C.) from a single lot of whites. Cakes were baked at the end of 1, 3, 5, 7, 9, 11, 24, 48, and 54 weeks. The data obtained are summarized in Table 3 and Figures 4 and 5.

TABLE 3

Influence of Freezing and Storage Temperatures on the Value of Egg White for Making Angel Food Cakes

Freezing and storage temperature		No. of trials	Cake (Average of 9)	
°F.	°C.		Volume (ml.)	Score [d]
Fresh (controls)		8	603	74
10	—12	9	581	72
0	—18	9	572	73
—10	—23	9	560	73
—20	—29	9	568	76
—50	—46	9	569	76

[d] Highest possible score 100.

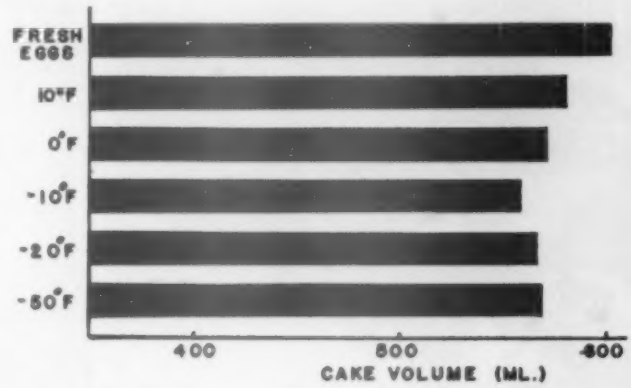

FIG. 4. Influence of freezing and storage temperatures on the value of egg white for making angel food cake.

The fresh egg white produced slightly better cake volume than the frozen whites. There was no appreciable difference in the volume or score of cakes produced with white which had been frozen at temperatures ranging from 10° F. (—12° C.) to —50° F. (—46° C.) and held for periods ranging from 1 to 54 weeks.

SUMMARY

Egg white pasteurized at 134° F. (57° C.) for 4 minutes produced undesirable angel food cakes unless the standard beating procedure was modified.

Satisfactory cakes, but not of optimum quality, were produced with pasteurized white when the beating speed and time were practically doubled during first beating and meringue stage, and then reduced correspondingly during the preparation of the batter.

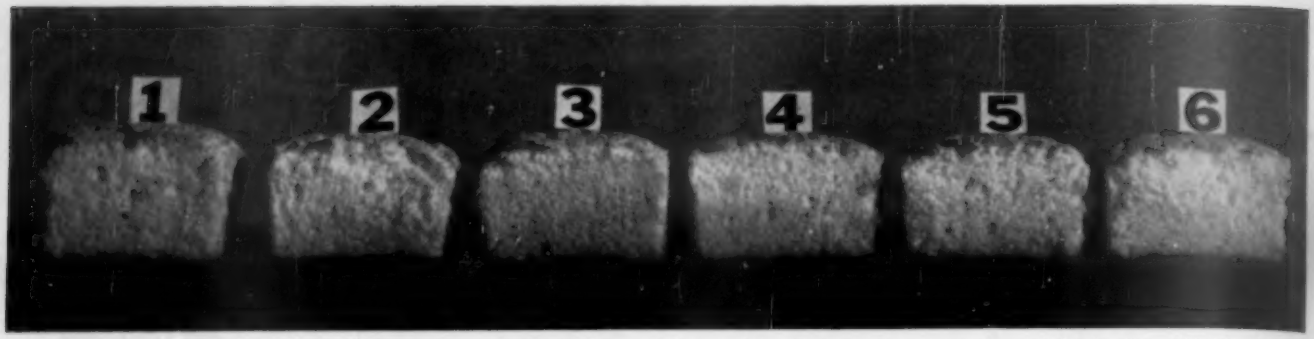

FIG. 5. Influence of freezing and storage temperatures of egg white on the volume and texture of angel food cake. (1) Fresh egg control, (2) stored at 10° F., (3) 0° F., (4) —10° F., (5) —20° F., (6) —50° F., respectively.

Cakes made from frozen egg white had nearly as good cake volumes as those produced from fresh egg whites. Judging scores were as good, or better with the frozen whites.

Freshly frozen egg white produced as good cakes as that which had been frozen as long as 24 weeks.

Egg white frozen and stored at 10°, 0°, —10°, and —20° F. (—12°, —18°, —23°, and —29° C.) for as long as 54 weeks produced angel food cakes of similar volume and score.

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Notes and Letters

The Estimation of Polygalacturonase (Pectinase) Activity

In a recent paper, Bell, Etchells, and Jones *(1)* describe a method for the estimation of the polygalacturonase activity which develops in brines during the pickling of cucumbers, in which the drop in viscosity is expressed as a percentage of the total drop. During the past two years a method of assay of commercial pectin-degrading enzymes has been used in this laboratory depending upon the fact that the percentage loss in viscosity/log amount enzyme is a linear function. The data of Bell, Etchells, and Jones *(1)* gives similar relationship (e.g. when the data in their Table 1, page 160 of their paper, are replotted), and it may be seen that doubling the concentration of the enzyme results in an increase of approximately 8.5 percent in the percentage loss in viscosity. In other words, if two preparations gave reductions in viscosity of 48.5 percent and 40 percent, the ratios of their activities would not be 48.5 to 40.0, but rather 2 to 1.

Therefore, to assess the activity of pectic enzyme preparations viscometrically, this logarithmic relationship must be taken into account. A revaluation of the data of Bell, Etchells, and Jones according to this correct activity relationship results in comparative activities which are different from those presented in the above paper.

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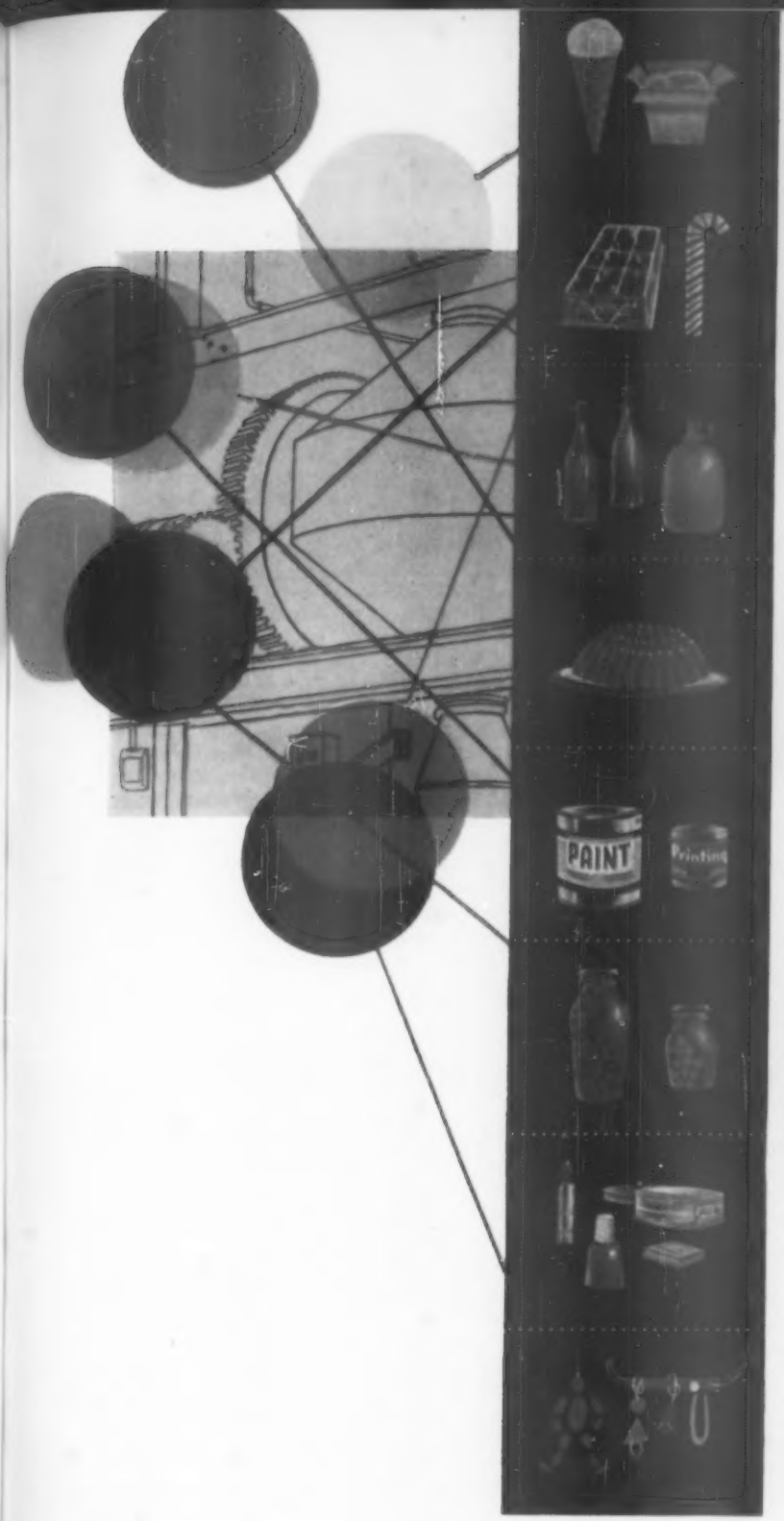
August 17, 1950.

LITERATURE CITED

1. BELL, T. A., ETCHELLS, J. L., JONES, I. P. Softening of cucumber salt stock in relation to polygalacturonase activity. *Food Technol.,* **4**, 157 (1950).



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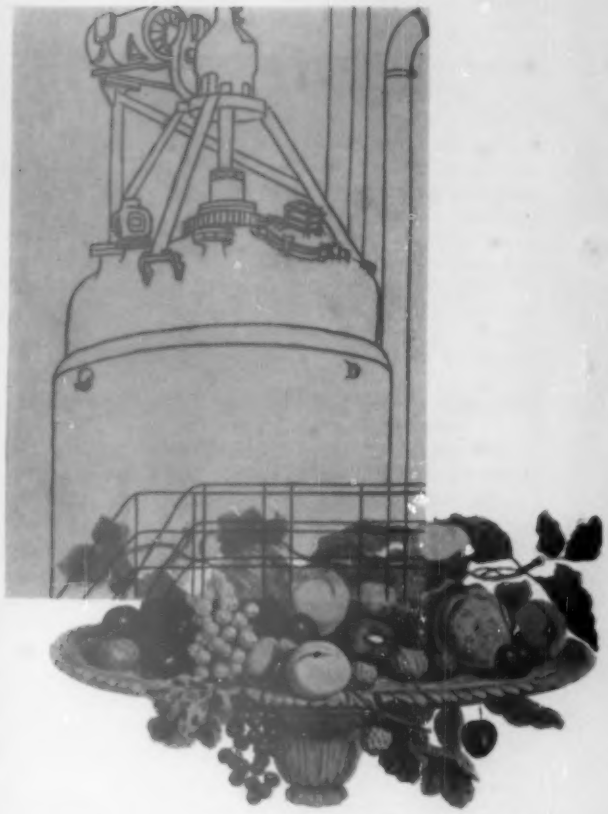

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Selected Abstracts

ANALYTICAL METHODS

Quantitative paper chromatography. I. Separation and gravimetric determination of thallium.

ANDERSON, J. R. A., AND LEDERER, M. *Anal. Chim. Acta., 4,* 513-16 (1950).

Separation and gravimetric determination of thallium. Using butanol saturated with NH_4Cl as solvent, trivalent thallium can be separated quantitatively from Fe, Cu, Ni and Co by a new technique of paper chromatography which employs strips of paper pulp.

An improved method of detecting sugars on paper chromatograms.

NOVELLE, L. Nature, *166,* 745-6 (1950).

Detection of sugars on paper chromatogram by use of β-naphthylamine spray. Fructose gives a bright yellow spot, pentoses give pink-red spots and aldohexoses give light brown spots.

Estimation of reducing sugars in starch hydrolyzates by paper chromatography.

SHU, P. *Canadian J. Research, 28B,* 527-34 (1950).

A method for quantitative determination of glucose, maltose, and reducing dextrins in starch hydrolyzates is described. The components are separated by paper chromatography and determined colorimetrically by measurement of their reducing power with alkaline copper and arseno-molybdate reagents. The probable error of the determinations with the use of this method is about 2%. A compact apparatus was designed, suitable for handling a large number of samples in the chromatographic separation. It is particularly useful for compounds with low R_f values.

Separation of starch and gluten. V. Problems in wheat starch manufacture arising from flour pentosans.

CLENDENNING, K. A., AND WRIGHT, D. E. *Canadian J. Research, 28F,* 390-400 (1950).

The voluminous starch sludge or "squeegee" fraction, obtained with all known methods of wheat starch manufacture, consists of strongly hydrated pentosan masses in which starch granules are embedded. The pentosans of the bran and endosperm both contribute to the formation of these heterogeneous complexes. The "squeegee" starch fraction is not abolished by allowing the crude starch slurries to ferment, by exposing them briefly to mildly acid or alkaline solutions, or to C_2-C_{12} monohydric alcohols. The pentosan and protein contents of this low quality starch fraction may be greatly reduced by 24-48 hr. digestion in dilute acid at pH 2.0 before recovering the starch on the centrifuge.

Determination of starch and amylose in vegetables. Application to peas.

MCCREADY, R. M., GUGGOLZ, J., SILVIERA, V., OWENS, H. S. *Anal. Chem., 22,* 1156-8 (1950).

Sugars are extracted from peas with 80% ethyl alcohol and the starch is solubilized with diluted perchloric acid. Starch is then determined colorimetrically, without previous acid hydrolysis, by means of the sugar-anthrone-sulfuric acid reaction. Amylose is estimated colorimetrically with iodine. Both total starch and amylose can be determined in about 30 minutes in sugar-free solutions. The accuracy and precision are at least equal to those of standard methods involving acid hydrolysis. Analyses of smooth and wrinkled dried peas and fresh peas are presented.

Soluble carbohydrates of fruit plants.

BRADFIELD, A. E., AND FLOOD, A. E. *Nature, 166,* 264-5 (1950).

Soluble carbohydrates of apple and plum plants have been under investigation using paper chromatography as a method. The soluble carbohydrate fraction contained fructose, glucose, sucrose, sorbitol and also raffiinose and stachyose.

Limits of the detection of glycols in glycerol by the method of Kroller.

NEU, R. *Pharmazie, 5,* 217-18 (1950); *J. Am. Oil Chemists Soc., 27,* 428 (1950).

The lower limits for the detection of glycols in glycerol are: ethylene glycol, 0.4%; 1, 2-propylene glycol, 1.2%, 1, 4-butylene glycol, 2%. The reaction can probably also be used for 1, 3-trimethylene glycol.

Microbiological determination of sulfur in yeast.

MCMANUS, D. K., SCHULTZ, A. S., AND MAYNARD, W. E. *Anal. Chem., 22,* 1187-90 (1950). A microbiological assay method for the determination of total sulfur in yeast is described in which *Torula utilis* is the test organism and methionine is the reference standard for sulfur. The microbiological assay results on yeast are compared with results from four known chemical methods. The method may be applicable to other substances. Results on whole soybean flour, defatted soybean flour, and vitamin-free casein indicate that the selection of the reference standard is important.

Copper contamination and ascorbic acid loss in Waring Blendor.

LAMDEN, M. P. *Anal. Chem., 22,* 1139-41 (1950).

The Waring Blendor, which has extensive usage in food analysis, vitamin assays, and biochemical preparations, is regarded with disfavor by some workers. Loss in ascorbic acid and loss in enzyme activity during blending have been cited. Adverse results may arise from use of containers having worn chrome plating on their blending assemblies. Copper dissolving from exposed brass parts may catalyze the loss of ascorbic acid in solutions during blending or create false high results in the determination of the copper content of certain foodstuffs. Inhibition of the destructive effect of copper dissolved from the blending assembly on ascorbic acid is discussed.

BIOLOGICAL SCIENCES

BIOCHEMISTRY

Effects of vitamin B_{12} on thiouracil action in rats.

MEITES, J. *Proc. Soc. Exptl. Biol. Med., 75,* 193-5 (1950).

The effects of crystalline vitamin B_{12} on thiouracil action was determined in immature female rats for a 30-day period. The vitamin completely counteracted the growth-inhibiting action of thiouracil, and this was accompanied by a considerable increase in food consumption. Although vitamin B_{12} decreased the thyroid hypertrophy induced by thiouracil, the uptake of radioactive iodine (I^{131}) by the thyroids was even less than in the rats which received thiouracil only. It is suggested that vitamin B_{12} may be able to induce normal growth in hypothyroid rats.

Vitamin B_{12}. I. The relation between vitamins B_{12} and B_{12b}.

VEER, W. L. C., EDELHAUSEN, J. H., WIJMENGA, H. G., AND LENS, G. *Biochim. Biophys. Acta., 6,* 225-8 (1950).

In mildly acid solutions, kept in the dark, the spectrum of vitamin B_{12} remains unchanged; on exposure to light, vitamin B_{12b} is rapidly formed. The spectrum of an irradiated solution, ($pH \geq 4$), subsequently kept in the dark, shows a slow reversal to that of vitamin B_{12}. When KCN is added to a solution of vitamin B_{12b}, rapid shift in the spectrum, from vitamin B_{12b} to vitamin B_{12}, can be observed.

Effects of vitamin B_{12} on normal thyroid function in rats.

MEITES, J. *Proc. Soc. Exptl. Biol. Med., 75,* 195-7 (1950).

Crystalline vitamin B_{12} was fed to immature rats of both sexes in order to determine whether the vitamin could alter thyroid function in normal or thyro-protein-treated rats. The growth rate of the rats supplemented with the vitamin was increased above that of the normal or thyroprotein-treated controls, but there was no significant effect on thyroid weight or uptake of I^{131}. It is concluded that vitamin B_{12} does not alter normal thyroid activity in rats.

NUTRITION

Sex differences in weight-stimulating effect of B_{12} in rats on diets of varying composition.

McCollum, E. B., and Chow, B. F. *Proc. Soc. Exptl. Biol. Med., 75,* 20-3 (1950).

Administration of B_{12} to rats from B_{12} depleted mothers produced increases in weight gains in both sexes on all of the rations studied. When the calories are derived primarily from carbohydrate, the vitamin exerts a greater effect on the weight gain in young female rats than in their littermate males. The stimulating effect of B_{12} was more marked in females on high carbohydrate diets than in littermate sisters on like diets except that fat replaces part or all of the carbohydrate.

Cobalt in the nutrition of sheep.

Keener, Percival, G. P., Ellis, G. H., and Beeson, K. C. *J. An. Sci., 9,* 404 (1950); *Vet. Med., 45,* 406 (1950).

An experiment was carried out with sheep in an attempt to learn more about the role of cobalt in the nutrition of ruminants. Every animal on the cobalt deficient diet developed marked symptoms. Those receiving cobalt supplements remained healthy. Those animals on a cobalt supplement gained 7 times as much weight. It was found both cobalt sulfate and cobalt carbonate relieved the deficiency when administered orally.

ENGINEERING

Fermentation. (Part of chemical engineering unit process review.)

Lee, S. B. *Ind. Eng. Chem., 42,* 1672-87 (1950).

A quick resume of the field with 333 references are included covering alcohols, microbiological amylases, acetone and butyl alcohol, 2, 3-butanediol, citric, lactic and other organic acids. Microbiological production of vitamins such as riboflavin, yeast production and antibiotics (penicillin, streptomycin, chloromycetin, aureomycin, bacitracin, neomycin, polymyxin, subtilin, tyrothricin, and terramycin). The growth of the proper microorganism, substrates, trace materials (particularly inhibitory effects) sterilization, fermentation equipment and the actual fermentation are treated as unit process factors.

FOOD PRODUCTS

FLAVORS

Vanillas as antioxidants in powdered ice cream mixes.

Pyenson, H., and Tracy, P. H. *J. Dairy Sci., 23,* 815-19 (1950).

Studies were made of nine vanillas and four vanilla compounds in powdered ice cream mixes held for one year at room temperature. The products used represented five different manufacturers of vanillas or vanilla compounds. Changes in the oxygen concentration of the headspace gas and palatability studies indicated that these vanillas and vanilla compounds have antioxygenic properties in powdered ice cream mixes. The addition of these vanillas or possibly the vanilla compounds would serve a two-fold purpose in powdered ice cream mixes, i.e., as flavoring and as an antioxidant.

FRUIT AND FRUIT JUICES

The retention of ascorbic acid in preserved fruits. II.

Crang, A., and Sturdy, M. *J. Sci. Food Agric., 1,* 252-4 (1950).

Further comparisons of domestic methods of preserving fruits have been made. The ascorbic acid retained in freshly-made jam from the three kinds of fruit was greatest in blackcurrant (70%), next in gooseberry (66%) and least in strawberry (55%), and after storage for one year the amounts were 30%, 28% and 17% respectively. The absolute values for ascorbic acid after one year's storage were approximately 6 mg./100 g. both for strawberry and gooseberry jams and 25 mg./100 g. for blackcurrant jam. The loss of this vitamin in making blackcurrant jelly varied considerably with the extraction methods used but was greater than in making jam. The ascorbic acid contents, before and after cooking, of three kinds of fruit preserved with sodium metabisulphite were compared with those of corresponding heat-processed fruits. The heat-processed

(Continued on page 16)

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Institute of Food Technologists Eleventh Annual Meeting

JUNE 17-21, 1951

Courtesy New York Convention and Visitors Bureau

Program: The full program of the eleventh annual meeting of the Institute of Food Technologists to be held in New York, June 17-21, 1951, will be published in the next (May) issue of FOOD TECHNOLOGY. Abstracts of all papers will be included. Thus for the first time the Institute's official publication will carry a complete record of the abstracts of papers in advance of presentation.

Assisting Dr. Charles N. Frey and his Program Committee in the organization of the technical part of the program has been a group of prominent members of the Institute of Food Technologists, each of whom has accepted responsibilities for a specific group of papers. These names, and their respective topics, follow: R. T. Bohn on Cereal Technology; B. S. Clark on Modern Tinplate and its Use in Packaging Food Products; L. E. Clifcorn on Food Technology and the Food Industry; A. L. Elder on Instrumentation and Control Methods in Food Processing; C. R. Fellers on Fish Products; G. Garnatz on Consumer Acceptance Testing; D. B. Hand on Low Temperature Evaporation of Food Products; R. A. Isker on Military Subsistence; M. B. Jacobs on Flavor; H. R. Kraybill on Recent Developments in the Meat Industry; A. G. Olsen on Plant Control Methods. Product Protection. Life Expectancy of the Product; B. L. Oser on Sanitation and Chemicals in Foods; M. E. Pennington on The Transportation of Foods; B. E. Proctor on Food Preservation; E. H. Wiegand on Frozen Foods.

Exhibits: Industrial exhibits prepared by about fifty major companies offering products, processes, and services to the food industry will be installed on the mezzanine floor of the Hotel New Yorker for the period of the meeting. More than 50 exhibit booths have been set aside in the north ballroom and adjacent areas on the mezzanine floor adjoining the main ballroom, where scientific sessions will be held.

The exhibition rooms will be open for inspection by food technologists on Sunday afternoon, June 17th. On the next 3 days, exhibits will be open from 8:30 A. M. to 7:00 P. M. This will give ample opportunity for the